

ZARETSKAYA, I.I.; SORKINA, T.I.; TORGOV, I.V.

Condensation of 1-vinyl-6-methoxy-3,4-dihydronaphthalene with  
2,4-dimethyl- $\Delta^2$ -cyclopentene-1,5-dione. Izv. AN SSSR. Ser. khim.  
no.6:1058-1061 '65. (MIRA 18:6)

1. Institut khimii prirodnykh soyedineniy AN SSSR.

KAZARNOVSKIY, Ya.S.; KOLODEYEV, I.P.; SORKINA, Ye.M.; IRLIN, A.L.  
SOLNTSEVA, L.N.

Oxidative thermal pyrolysis of hydrocarbon gases to acetylene.  
Khim. prom. no. 7:547-551 O-N '60. (MIRA 13:12)  
(Hydrocarbons) (Acetylene)

SOKINA, Ye.Z.

Clinical aspects of early primary tuberculosis in school children.  
Probl. tuberk., Moskva no. 3:37-41 May-June 1952. (CLML 22:4)

1. Candidate Medical Sciences. 2. Of the Dispensary Sector (Head  
-- Prof. S. Ye. Nezlin), Institute of Tuberculosis of the Academy  
of Medical Sciences USSR (Director -- Z. A. Lebedeva).

SORKINA, Yu. A.; CHERNOV, V. A.

Antineoplastic activity of some piperazine derivatives. Vop. onk.  
(MIRA 15:2)  
8 no.1:77-84 '62.

1. Iz laboratorii eksperimental'noy khimioterapii opukholey  
(rukov. - kand. biol. nauk V. A. Chernov) otdela khimioterapii  
(rukov. - prof. G. N. Pershin) Vsesoyuznogo nauchno issledovatel'-  
skogo khimiko-farmatsevticheskogo instituta im. S. Ordzhonikidze.

(PIPERAZINE) (CYTOTOXIC DRUGS)

CHERNOV, V. A.; PISKOV, V. B.; SORKINA, Yu. A.; LYTKINA, L. G.;  
LYTKINA, V. B.

Antiblastic activity of compounds containing an ethylene bond  
joined to carbonyl. Vop. onk. 8 no.5:24-32 '62.  
(MIRA 15:7)

1. Iz laboratorii eksperimental'noy khimioterapii opukholey  
Vsesoyuznogo nauchno-issledovatel'skogo khimiko-farmatsevti-  
cheskogo instituta im. S. Ordzhonikidze i laboratorii radiologii  
instituta meditsinskoy i biologicheskoy khimii AMN SSSR.  
Adres avtorov: Moskva, G-21, ul. Zubovskaya, d. 7, Vsesoyuznyy  
nauchno-issledovatel'skiy khimiko-farmatsevticheskiy institut  
imeni S. Ordzhonikidze.

(ETHYLENE) (CARBONYL GROUP) (CYTOTOXIC DRUGS)

SORKINA-FINKEL', L.I.

\*From the pages of foreign biological and agricultural publications. Agrobiologia no.4:637-638 Jl-Ag '63. (MIRA 16:9)  
(No subject headings)

NEYMAN, N.F.; SORKINA-FINKEL<sup>1</sup>, L.I.

From the pages of foreign biological and agricultural publications. Agrobiologija no.6:950-952 N-D '63.  
(MIRA 17:2)

HAYMAN, N.F.; SOKINA-FINKEL', L.I.

From the pages of foreign biological and agricultural publications. Agrobiologija no.1:158-159 Ja-F '64  
(MIRA 17:8)

SORKINA-FRENKEL', Ye.

From the pages of foreign biological and agricultural publications.  
Agrobiologiya no.5:797-798 S-0 '62. (MIKA 15:11)  
(Bibliography--Agriculture)

NEYMAN, N.F.; SORKINA-FINKEL', L.I.

From the pages of foreign biological and agricultural publications.  
Agrobiologija no.2:317-319 Mr-Ap '63. (MIRA 16:7)  
(Bibliography--Agricultural research)

NEYMAN, N.F.; SORKINA-FINKEL', L.I.

From the pages of foreign biological and agricultural publications.  
Agrobiologiya no.3:476 My-Je '63. (MIRA 16:7)  
(No subject heading)

NEYMAN, N.F.; SOKKINA-FINKEL', L.I.

From the pages of foreign biological and agricultural publications.  
Agrobiologija no. 3:475-477 My-Je '64. (MIRA 17:7)

NEYMAN, N.F.; SORKINA-FINKEL', L.I.

From the pages of foreign biological and agricultural publications.  
Agrobiologiiia no.4:637 Jl-Ag '64. (MIRA 17:12)

SORKO, L.M.

The reaction  $p + p \rightarrow J/\psi + p + p$  in the 400-660 Mev. range. Zhur. ekspl. i teor. fiz. 30 no. 2:296-303 F '56.  
(Nuclear reactions)

SORLIN, Arsene

Variations of the lifetime of free minority carriers in germanium  
considered as a function of pressure. Bul Ac Pol mat 8 no.1:71-75  
'60. (EEAI 9:11)

1. Instytut Fizyki, PAN. Presente par A.Soltan.  
(Germanium)  
(Semiconductors)  
(Electric conductivity)  
(Pressure)

EXCERPT AND PROPERTIES

Capacity phenomena displayed at mercury capillary electrodes I. Hlavovský, F. Šem, and J. Forejt (Charles Univ., Prague, ČSSR) *Collection Czechoslov. Chem. Commun.*, 12, 11-38 (1947) (in English).

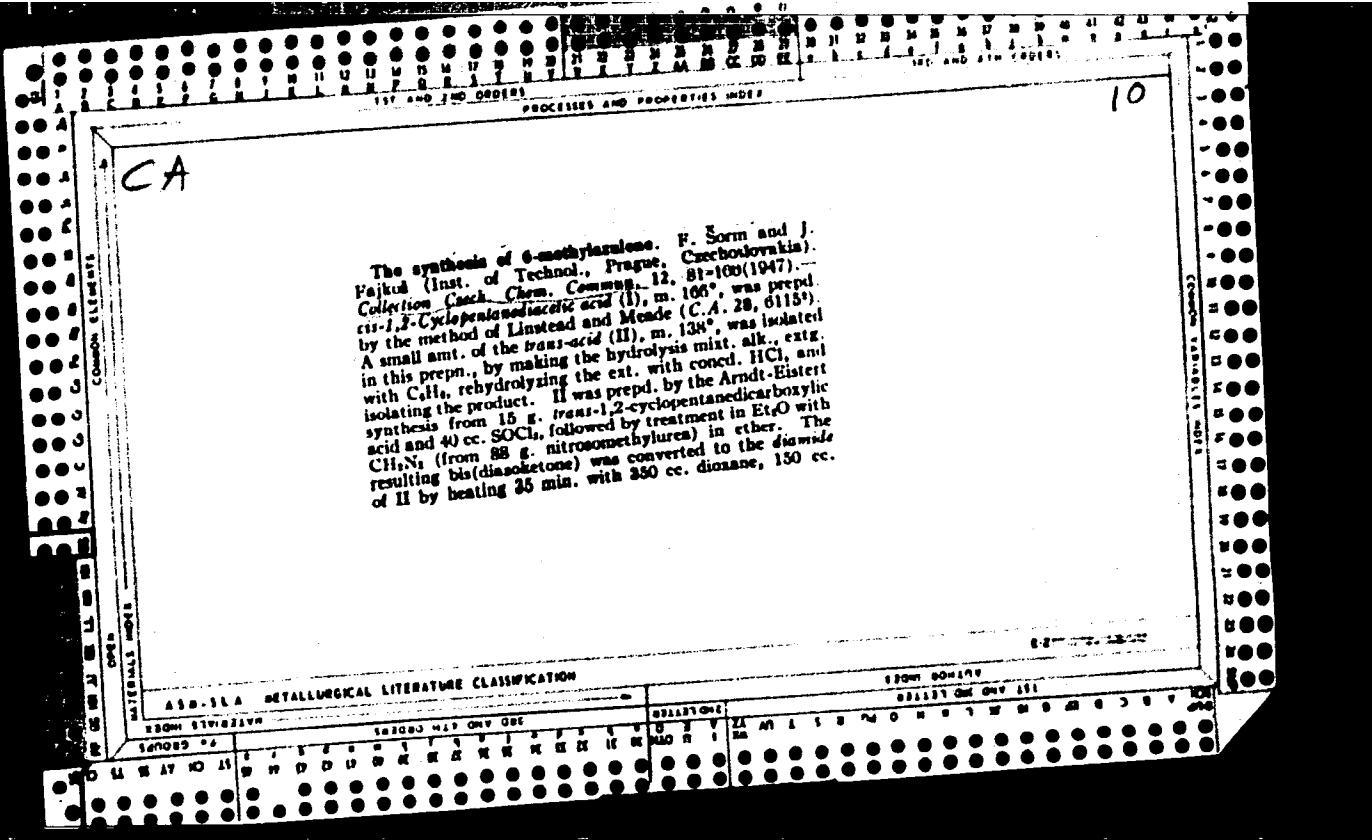
The capillary electrodes used were the dropping Hg electrode and the streaming Hg electrode. The latter produces a continuously renewed uniform surface of Hg of suitable dimensions because the Hg is forced in a continu-

ous stream in fine jet and upwards through 1.8 mm. of soln. For voltammetric studies this has the advantage that complications arising from a growing Hg drop are eliminated. Results obtained with both capillary electrodes were essentially the same. Studies were made with the polarograph and with a cathode-ray oscillograph. The latter could be connected either to show changes in the pattern of a square wave produced by the phenomena at the capillary electrode directly, or to give only the deriv. of this curve. It was found that certain relatively insol. substances, e.g., pyridine in alkali, butyric acid in acid, and ether in any electrolyte soln., produced a peculiar charging effect by their adsorption on the electrodes. This became apparent as a time-lag on the oscillographic curves or as a diminished condenser current on the polarographic curves which ceased at a characteristic voltage. It was concluded that this phenomenon is caused by a film of the nonelectrolyte adsorbed on the electrode. This film can break up suddenly and can also be rebuilt at speeds greater than 0.01/sec.; it has no measurable resistance; it hinders the electroreduction of  $Pb^{++}$ ,  $Cd^{++}$ , or nitrobenzene, but does NOT interfere with that of  $Tl^+$ . These results are thought to indicate that only one electron is obtained from the electrode by the bivalent ion at any one time and that a subsequent dismutation in soln.:  $2Pb^+ \rightarrow Pb + Pb^{++}$  is hindered by the adsorbed film.

Otto H. Müller

## ABE-SLA METALLURGICAL LITERATURE CLASSIFICATION

SEARCHED		SERIALIZED		INDEXED		FILED	
SEARCHED	SEARCHED	SERIALIZED	SERIALIZED	INDEXED	INDEXED	FILED	FILED
SEARCHED	SEARCHED	SERIALIZED	SERIALIZED	INDEXED	INDEXED	FILED	FILED

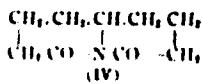
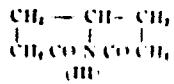


CA

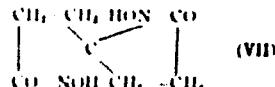
A simple synthesis of racemic hygrine. F. Sorm (Society for Chemical and Metallurgical Production, Prague-Vysocany). Collection of Czechoslov. Chem. Commun., 12, 245-48(1947); cf. U.S. 2,611,148 - *1-Methyl-2-acetyl-pyrrole* (I), obtained in 5.7 g. yield from 20 g. *1-methylpyrrole* (II) and 1 g. Cu bronze at 100° and dropwise addn. over 30 min. of a mixt. of 8.7 g. freshly distd. diazocetone and 5 g. II, b.p. 100-111°; 17 g. II was recovered from the reaction mixt.; I is very unstable; the semicarbazone m. 107° but cannot be purified due to instability. Freshly distd. I (6.3 g.) in 50 cc. HOAc and 0.4 g. PtO<sub>2</sub> (prepd. according to Adams) absorbed at 100 mm. pressure in 7 hrs. 1900 cc. H<sub>2</sub> (0° and 700 mm.) (2030 cc. calcd.). The filtrate turned intensely red after standing 1 week at room temp., giving 3.8 g. (60%) *hygrine (1-methyl-2-acetylpyrrolidine)* (III), b.p. 88°. The picrate of III m. 155° (from EtOH); this sharp m. p. is the same as found for the picrate from natural III, but lower than the indistinct m. p. reported by Hess (C.A. 8, 127, 934). The oxime of III m. 125° (same as Hess) but the picrate of the oxime m. 166° (Hess, 163°).

ASG-SLA METALLURGICAL LITERATURE CLASSIFICATION		E-2-10-22-222	
SEARCHED	INDEXED	FILED	SEARCHED
SERIALIZED	FILED	SEARCHED	SEARCHED
FILED	SEARCHED	SEARCHED	SEARCHED
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JAN 1999 MAR 1999 JUN 1999 SEP 1999 DEC 1999		JULY 1999 AUG 1999 NOV 1999 FEB 2000 MARCH 2000 JUNE 2000	

Symmetrical amino dicarboxylic acids and their cyclization. R. Lukeš and F. Ševčík (United Chemical and Metallurgical Manufacturers, Prague-Vysočany). *Czechoslovak Chem. Commun.* 12, 278-91 (1947); cf. *C.A.* 47, 2041; 28, 5825. Sym. amino dicarboxylic acids, specifically  $\gamma$ -aspartic acid (**I**) and  $\beta$ -aspartic acid (**II**), were sought for subsequent ring closure to the bicyclic dioxetans.



*D,L*- $\beta$ - $\gamma$ -ketopeptidic ester (**V**), m. 38°, (30 g.) in 400 cc. 90% EtOH and 1.5 g. PbO (according to Adams) and 0.2 g.  $\text{FeCl}_3$  ( $\text{H}_2\text{O}$ ) dissolved in 150 mm. 30%  $\text{H}_2\text{SO}_4$  (0° and 200 mm.) (91.40 cc. added) to give 40% *D,L*-2-pyrrolidone-5-propionate (**VI**) and a little di- $\beta$ - $\gamma$ -ketopimelate, m. 38° (from  $\text{CaH}_2$ ), (1, obtained from 10 g. **VI** and 300 cc.  $N\text{H}_2\text{SO}_4$  at 100° for 1 hr., m. 180°. Electrolytic reduction of 118 g. **V**, in 550 cc. 30%  $\text{H}_2\text{SO}_4$  with Pb electrodes and cathode density of 1 amp. sq. dm., gave only 11 g. 1, di- $\beta$ -pimelite, and a cryst. product (**VII**,  $\text{C}_{11}\text{H}_{16}\text{N}_2$ , possibly a

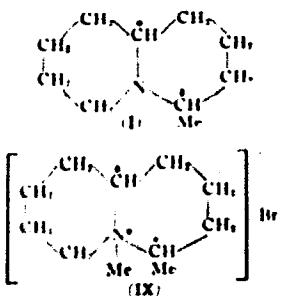


which blackened at 230° but did not m. below 275° (little prisms from  $\text{H}_2\text{O}$ ). Diacetate, from 0.5 g. **VII** and 10 cc.  $\text{Ac}_2\text{O}$  under reflux for 2 hrs., colorless needles, m. 190°.

(from EtOH). Reduction of 174 g.  $\gamma$ -ketopimelate acid with 280 g.  $\text{HCNHN}_3$  and 190 g.  $\text{HCO}_2\text{H}$  95 hrs., with the temp. rising gradually from 150° to 210° gave a resin. Rktm. with boiling 10%  $\text{H}_2\text{SO}_4$ , 3 hrs., addn. of 300 cc. EtOH gave a ppt. of 88 g. 1, m. 100°, and some 2-pyrrolidone-5-propionate, and (**VIII**), m. 128°. **VIII**, colorless crystals, was also obtained by heating 1 g. 1 at 200° 2 hrs. and extg. with 200 cc.  $\text{CaH}_2$ . Heating 1 in a test tube in a bare flame 1.5 min. and then extg. with hot MeOH gave 52% **III** as needles several cm. long, m. 181° (from EtOH, then  $\text{CaH}_2$ ). Reduction of 28 g.  $\delta$ -ketovetic acid, m. 110.5°, with 120 g.  $\text{HCNHN}_3$  and 80 g.  $\text{HCO}_2\text{H}$  10 hrs. at 175°-200°, gave 42 g. 1, m. 102° (from EtOH). Heating 1 in an open tube at 200° 2 hrs. gave 2-pyrrolidone-6-butyne acid (**IX**), m. 110° (from  $\text{CaH}_2$ ). Further heating of **IX**, even distn. or refluxing 3 hrs. with 10 parts  $\text{SOCl}_2$ , gave no change to the desired **IV**. The dropwise addn. of a soln. of 0.5 g.  $\text{Mg}$ , 30 cc.  $\text{Et}_2\text{O}$ , and 2.53 g.  $\text{PbCl}_2\text{Cl}$  to 0.7 g. **III** in the min. amt. of  $\text{CaH}_2$  gave an initial ppt.; ice and 10 cc. concd. HCl were added after 24 hrs. and a product,  $\text{C}_{11}\text{H}_{16}\text{N}_2$ , m. 90° (colorless needles from  $\text{EtOAc}$ ), was obtained, presumably 2,2-divinyldipyrrolidone-5-propionate, and. Drying at elevated temp. produced a loss of  $\text{H}_2\text{O}$  and formation of a vitreous mass. Reduction of 7 g. **III** in 60 cc. 30%  $\text{H}_2\text{SO}_4$  electrolytically with a Pb cathode for 60 amperes (10.8 v.d.) gave only a little pyrrolidine-2-carboxylic acid, m. 257° (cf. *Czech. Obor.* 18, 102 (1943)). No monodivinylpyrrolidine was found and 4 g. **III** was recovered. A sample of **V** which had been stored 1 year contained 10% of an ether insol. product, cryst. from  $\text{H}_2\text{O}$  as colorless strongly refractive prisms, darkening at 230° and analyzing as **VII**. The failure of **IX** to give **IV**, in contrast to the behavior of **VIII**, is attributed to a functional difference in the amide groupings in these 2 compds.

John W. Green

Syntheses in the allotropane series. R. Lukes and  
F. Smetanova, *Czechoslov. Chem. Commun.*, 12,  
350-57 (1947).—A method used previously by Lukes and  
Smetackova (*C. A.* 39, 7941; 38, 5825) for the synthesis  
of  $\alpha$ -substituted tertiary piperidines has been applied to  
the prepn. of allotropane (I).  $\text{CH}_3\text{CH}(\text{CH}_2\text{CH}_2\text{MgBr})$  and  
 $\text{C}_6\text{H}_5\text{O}$  gave 58%  $\text{CH}_2=\text{CHCH}_2\text{CH}_2\text{CH}_2\text{OH}$  (III), b. 135°.



II gave 51%  $\text{CH}_2=\text{CHCH}_2\text{CH}_2\text{CH}_2\text{Br}$  (III) according  
to Juvula (*C. A.* 25, 4844). To 240 g. III, 40 g. Mg, and  
850 cc. Et<sub>2</sub>O was added 113 g. 1-methyl-2-piperidone in  
Et<sub>2</sub>O. The mixt. was decompd. the following day by ice  
and Ba(OH)<sub>2</sub>, steam distil., and the distillate neutralized  
with 100 cc. 5 N HCl and evapd. in vacuo; on cooling crys-

tals sepd. from which was liberated 60 g. of the rather un-  
stable base, 1-methyl-2-(4-pentenyl)-1,4,5,6-tetrahydro-  
pyridine (IV), b.p. 107°. (Some 1-methyl-2,2-bis(4-  
pentenyl)piperidine was probably formed also.) To IV,  
HCl in 80 cc. HCl (1:1) was added 400 g. So granules,  
and reaction continued by adding 80-cc. portions of HCl  
(total 800 cc.) at intervals during 32 hrs.; neutralization  
by solid NaOH, steam distil., exact neutralization of the  
distillate by HCl, evapn. in vacuo, liberation of the base,  
and distn. gave 2 fractions, b<sub>1</sub> 116-43° and b<sub>2</sub> 144-62°.  
Redistn. of the latter gave 29 g., b<sub>2</sub> 146-7°, of 2-methyl-  
6-(4-methylamirobutyl)tetrahydropyran (V), did not ab-  
sorb Br, 1.02 moles active H (Zerewitinoff), d<sub>4</sub><sup>20</sup> 0.9635,  
n<sub>D</sub><sup>20</sup> 1.47512, n<sub>D</sub><sup>20</sup> 1.47778, m.p. 1.48-1.27, n<sub>D</sub><sup>20</sup> 1.48096, MD 55.90,  
MD calcd. 56.04. Possible structures other than IV,  
1-methyl-2-(4-hydroxypentyl)tetrahydropyridine, and  
 $\text{MeNH}(\text{CH}_2)_2\text{CH}(\text{OH})(\text{CH}_2)_2\text{CH}:\text{CH}_2$ , were ruled out.  
The formation of V is explained by a tautomeric ring oper-  
ing of the hydrate of IV to  $\text{MeNH}(\text{CH}_2)_2\text{CO}(\text{CH}_2)_2\text{CH}_2$ .

## A50-51A METALLURICAL LITERATURE CLASSIFICATION

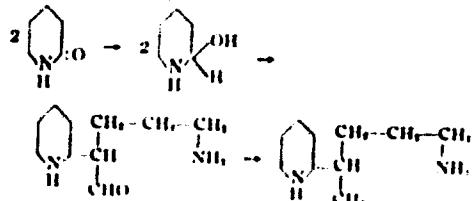
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3	W	H	D	D	P	M	R
4	W	H	D	D	P	M	R
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6	W	H	D	D	P	M	R

CA

## PROCESSES AND PROPERTIES INDEX

10

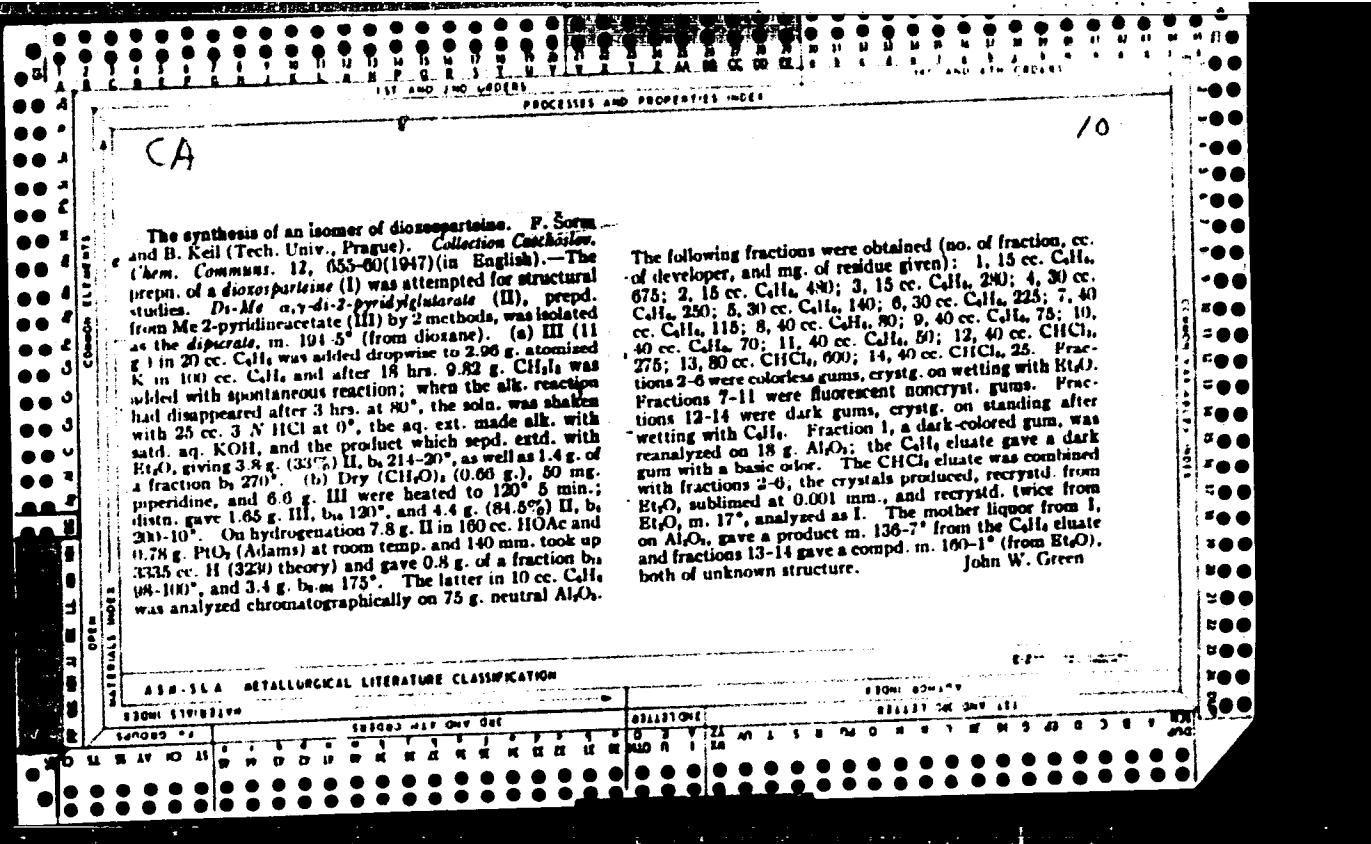
Action of propylmagnesium bromide on 2-pyrrolidone and 2-piperidone. R. Lukel, E. Sorm, and Z. Arnold (Ecole polytech., Prague). *Collection Czechoslov. Chem. Commun.*, 12, 641-6 (1947) (in French); cf. C.A. 35, 102.  $\text{PrMgBr}$  (I) was made to react with lactams containing an active H on the N atom. Mg (31 g.) and 164 g.  $\text{PrBr}$  in 500 cc.  $\text{Et}_2\text{O}$  were converted to I, 500 cc. xylylene added, and the  $\text{Et}_2\text{O}$  distilled; 29 g. 2-pyrrolidone in 60 cc. xylylene was then added at 100° dropwise; more  $\text{Et}_2\text{O}$  distilled at 130°, and the mixt. kept at 140-5° 6 hrs. Addn. of II (II) and ice, then 700 g.  $\text{BaO}$ , neutralization of the steam distillate with 10%  $\text{HCl}$ , concn. to a small vol., and treatment with  $\text{KOH}$  gave the free bases. Two fractions were obtained from the  $\text{Et}_2\text{O}$  ext., 7.0 g. racemic  $\gamma$ -coniceine (III), m.p. 88.5°, and a possible dimer (III),  $\text{Cu}^{+}\text{In}_2\text{N}_3$ , m.p. 118-22°. II picrate, from  $\text{HCl}$  soln. with 1 equiv. Na picrate, m. 79° (from  $\text{Et}_2\text{O}$ ); II picromate, from the free base and picromic acid in  $\text{EtOH}$ , m. 131°; III picrate m. 101° (from  $\text{Et}_2\text{O}$ ). The high b.p. of III points to a dimeric structure; its formation is attributed to the reducing action of I. The provisional reaction is:



I, from 50 g. Mg, 250 cc.  $\text{PrBr}$  in 800 cc.  $\text{Et}_2\text{O}$ , and 200 cc. xylylene, was treated at 100° with 43.5 g. 2-pyrrolidone in 50 cc. xylylene; 3 fractions were obtained. 2-propylpiperidone (IV), m. 60.7°, 6 g. of a mixt. of depropylpiperidone (V), and a base,  $\text{Cu}^{+}\text{In}_2\text{N}_3$ , m. 125-35°. IV picrate m. 115.6°; the benzene/ $\text{NaOAc}$  m. 88.5°; both gave no depression in mixed m.p. with authentic compds. V and VI were sepd. by crystn. of the picrates in  $\text{EtOH}$ , both as yellow needles. V picrate m. 240°; VI picrate m. 167°. VI has 3 IV groups and a single double bond.

John W. Gaten

## ASA 514 METALLURGICAL LITERATURE CLASSIFICATION



SHORM, F.

F-2

USSR / Microbiology-Antibiosis and Symbiosis.  
Antibiotics

Abs Jour: Ref Zhur - Biol., No 6, 1958, 24135

Author : Gryunberger, D., Shormova, Z., Shorm, F.  
Inst : Not given  
Title : Effect of Albomycin on Oxidation Processes and  
Metabolism of Nucleic Acids in Staphylococcus  
Aureus and in Escherichia Coli.

Orig Pub: Biokhimya, 1947, 22, No 1-2, 2, 148-153

Abstract: Albomycin (I) in greater than bacteriostatic concentrations ( $10 \text{ Y/ml}$ ) does not inhibit endogenous respiration and oxidation of glucose, acetic, succinic and glutamic acids either in dormant or growing bacterial cells of Escherichia coli and Staphylococcus aureus. Exposure of growing cells of S. aureus in the period of logarithmic phase to

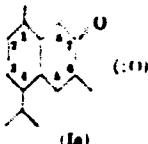
Card 1/2

**Polarographic reduction of heterocyclic compounds. II**  
P. Šorm and Z. Šornová. *Chem. Listy* 42, 82-7 (1948).  
In a study of systems similar to one of the components of  
codehydrogenase, the following compd.s were subjected to  
polarographic reduction: Pyridine-MeBr, trigonelline, niacin  
amide-MeBr, niacinotinamide-MeBr, 1-tetraethyl-  
chloro-3-carbamylpyridinium bromide, and cozymate. The  
varying behavior of the compd.s is related to their struc-  
tures. Where 2 waves are found, the 1st is assoc. with 1-  
electron reduction to a semiquinonoid compd., the 2nd  
to the reduction to a dihydropyridine deriv. M. Hudlický

*Terpenes. XII. Composition of the oil of hops.* F. Sorm, J. Mlečiva, and Z. Arnold (Tech. Univ., Prague). Collection Czechoslov. Chem. Commun. 14, 603-8 (1949) (in English); cf. C.A. 44, 1036, 5848c.—Oil of hops is examined to define the sesquiterpene fraction, which is separable as pure components by a chromatographic method. Lupulone (I) is identified as 3 $\alpha$ -methyl ketone. The previously reported nonanane-carboxylic acid is identified as pelargonic acid (III). It is present as esters of nonidentified acids. A C<sub>11</sub>H<sub>18</sub>O ketone is present in small amount in the higher-boiling fractions. Two cryst. triterpenes, C<sub>28</sub>H<sub>48</sub> (III), m. 34° (octahydroderiv., m. 37°), and a pristane (IV), m. 107°, n<sub>D</sub><sup>20</sup> 1.477, mol. wt. 412, are also present. It is concluded that the principal constituents of the Bohemian oil are myrcene, pelargonic esters, McCOCCHL<sub>10</sub>, and the triterpenes III and IV. XIII. *Sesquiterpenes from the essential oil of hops.* F. Sorm, J. Mlečiva, Z. Arnold, and J. Pliva. *Ibid.* 609-715.—The previously reported identity of humulene and  $\alpha$ -caryophyllene is studied. The C<sub>15</sub>H<sub>24</sub> sesquiterpene fraction of the oil is sep'd. chromatographically as the 3 components: natural farnesene (I), acyclic with 4 double bonds, 2 of which are conjugated; humulene (II), monocyclic with 3 double bonds; and a  $\beta$ -caryophyllene type (III), bicyclic with 1 double bond. The constitution of I proposed is 2,6-dimethyl-10-methylene-3,8,11-dodecatriene. XIV. *The Identity of humulene and  $\alpha$ -caryophyllene.* V. Herout, M. Štríbrná, J. Mlečiva, and F. Sorm. *Ibid.* 716-22.— $\alpha$ -Caryophyllene (I) obtained from oil of clover was isolated as a pure compl., bp 126°, d<sub>25</sub><sup>20</sup> 0.8600, n<sub>D</sub><sup>20</sup> 1.4920, n<sub>D</sub><sup>20</sup> 1.0 + 0.3° (CHCl<sub>3</sub>, + 0.20). Hexahydro deriv. of I, bp 118°, d<sub>25</sub><sup>20</sup> 0.8644, n<sub>D</sub><sup>20</sup> 1.4715.

d<sub>25</sub><sup>20</sup> = 0.8 + 0.1° (CHCl<sub>3</sub>, + 0.22); fumamate, m. 119°, 1-tridecide (from I and Ba(OH)<sub>2</sub>), m. 121°. The infrared spectra of I and its hexahydro deriv. are identical with those of humulene (II) of oil of hops and hexahydrohumulene (III). It is proposed that the name caryophyllene, which indicates bicyclic hydrocarbons, be replaced by "humulene" which is monocyclic. XV. *Composition of "humulene"* which is monocyclic. F. Sorm and V. Herout. *Ibid.* 723-40.—Acrone (I), C<sub>10</sub>H<sub>16</sub>O, and isocrone (II), the sesquiterpene diketones from oil of sweet flag, exist in emulsion equil. and are separable chromatographically. I with Pd-H yields acetoneone (III), C<sub>10</sub>H<sub>16</sub>O, a keto alc. (p-nitrophenylate, m. 131°); semicarbazone, amorphous. I with  $\alpha$ -HO(C<sub>2</sub>H<sub>5</sub>)<sub>2</sub> yields acetoneone (IV), C<sub>10</sub>H<sub>16</sub>O, m. 188°, cryst. as the lactone. The lactone from II, m. 127°, I and II do not react with KMnO<sub>4</sub>. I is demicarbazone, m. 211° (decompn.), regenerates a mixt. of I and II when hydrolyzed with (CuO)<sub>2</sub>. Zn-Hg reduces I to a mixt. (C<sub>11</sub>H<sub>20</sub> and C<sub>11</sub>H<sub>18</sub>) (V), and Pt(O<sub>2</sub>)<sub>2</sub>-H reduces V to acetone (VI), bp<sub>1</sub> 124-6°. Pt-C dehydrogenates V at 310° to cadalene (VII). LiAlH<sub>4</sub> in Et<sub>2</sub>O converts I to acordiol (VIII), amorphous, which is dehydrated with  $\alpha$ -bonds. IX with Pt(O<sub>2</sub>)<sub>2</sub>-H yields VI and Pt-C dehydrogenates IX to VII. The cadalene skeleton probably is obtained by rearrangement. I on NaOH fuses at 226° and yields piperitone and a doubly-unsatd. alc., C<sub>11</sub>H<sub>18</sub>O (X) (benzylthiuronium salt, m. 120°; its ester, bp<sub>1</sub>

130°). I with  $\text{PtCl}_6\text{-Hg}$  yields a tetrahydroderiv., b, (3)-J<sup>4</sup>. I with  $\text{MeMgI}$  yields methylsuccone (XII), m, 130°, which forms with  $\text{LiAlD}_3$  methylsuccind (XIII), m, 119°. XIII dehydrates to a doubly unsatd.  $\text{C}_{10}\text{H}_{16}$  (XIV), b, 132°. XIII with Pd-C dehydrogenates to 7-methylcadalene, m, 39.5°, and minor amts. of azulene. I appears to show an anomalous  $\text{CH}_3\text{I}_2$  reaction because an Ac grouping is not proved for I, and I thus far subjected to a Beckmann rearrangement does not form volatile acids or amines. The data indicate for I the formula Ia, where the



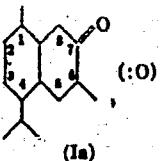
5-position is favored for the 2nd CO group. I with  $\text{BzLi}$  forms monobenzylideneacarone,  $\text{C}_{11}\text{H}_{10}\text{O}_2$ , m, 183-0°. The results indicate position 7 for the reactive carbonyl while the 2nd carbonyl, probably in position 5, is sterically hindered. The failure of the characteristic  $\text{FeCl}_3$  test for a  $\beta$ -diketone and ultraviolet spectra exclude  $\alpha$ - and  $\beta$ -diketone structures. The differences in the infrared spectra of acarone and tetrahydrcadalene suggest that I and II do not have the cadalene skeleton but form cadalene by rearrangement during dehydrogenation. N. T. P.

SOBH, E.

Terpenes. XII. Composition of the oil of hops. E. Šorm, J. Mleziva, and Z. Arnold (Tech. Univ., Prague). *Czechoslov. Chem. Commun.* 14, 683-8 (1949) (in English); cf. C.A. 44, 1036, 5848c.—Oil of hops is examd. to define the sesquiterpene fraction, which is separable as pure components by a chromatographic method. Luparone (I) is identified as Me nonyl ketone. The previously reported isononaniccarboxylic acid is identified as pelargonic acid (II). II is present as esters of nonidentified alcs. A  $C_{10}H_{16}O$  ketone is present in small amount in the higher-boiling fractions. Two cryst. triterpenes,  $C_{30}H_{50}$  (III), m. 34° (octahydro deriv., m. 37°), and a product (IV), m. 60°,  $\alpha_D^{25} -17^\circ$ , mol. wt. 412, are also present. It is concluded that the principal constituents of the Bohemian oil are myrcene, pelargonic esters,  $MeCOCH_2H_5$ , and the triterpenes III and IV. XIII. Sesquiterpenes from the essential oil of hops. F. Šorm, J. Mleziva, Z. Arnold, and J. Pliva. *Ibid.* 699-718.—The previously reported identity of humulene and  $\alpha$ -caryophyllene is studied. The  $C_{15}H_{24}$  sesquiterpene fraction of the oil is sepd. chromatographically as the 3 components: natural farnasene (I), acyclic with 4 double bonds, 2 of which are conjugated; humulene (II), monocyclic with 3 double bonds; and a  $\beta$ -caryophyllene type (III), bicyclic with 1 double bond. The constitution of I proposed is 2,6-dimethyl-10-methylene-2,6,11-dodecatriene. XIV. The identity of humulene and  $\alpha$ -caryophyllene. V. Herout, M. Streible, J. Mleziva, and F. Šorm. *Ibid.* 718-22.— $\alpha$ -Caryophyllene (I) obtained from oil of cloves was isolated as a pure compd., b.p. 126°,  $d_4^{20} 0.8980$ ,  $n_D^{20} 1.5923$ ,  $\alpha_D^{20} 1.0 \pm 0.3^\circ$  ( $CHCl_3$ , c 0.20). Hexahydro deriv. of I, b.p. 118°,  $d_4^{20} 0.8884$ ,  $n_D^{20} 1.4715$ .

$\alpha_D^{20} -0.8 \pm 0.3^\circ$  ( $CHCl_3$ , c 0.33); I nitrosite, m. 119°. I trioxide (from I and  $Br_2O_2H$ ), m. 131°. The infrared spectra of I and its hexahydro deriv. are identical with those of humulene (II) of oil of hops and hexahydrohumulene (III). It is proposed that the name caryophyllene, which indicates bicyclic hydrocarbons, be replaced by "humulene" which is monocyclic. XV. Constitution of acorone and isoacorone. I. P. Šorm and V. Herout. *Ibid.* 723-40.—Acorone (I),  $C_{15}H_{20}O$ , and isoacorone (II), the sesquiterpenic diketones from oil of sweet flag; exist in enolization equil. and are separable chromatographically. I with Pt-H yields acorolone (III),  $C_{15}H_{18}O_2$ , a keto alc. ( $p$ -nitrobenzoate, m. 133°); semicarbazone, amorphous). I with  $\alpha$ -HO $_2CC_2H_4CO_2H$  yields acoronolide (IV),  $C_{15}H_{20}O_3$ , m. 188°, cryst. as the lactone. The lactone from II, m. 137°. I and II do not react with  $KMnO_4$ . I disemicarbazone, m. 211° (decompn.), regenerates a mixt. of I and II when hydrolyzed with ( $CO_2H$ ). Zn-Hg reduces I to a mixt. ( $C_{15}H_{20}$  and  $C_{15}H_{18}O$ ) (V), and Pt-O $_2H$  reduces V to acrone (VI), b.p. 124-6°. Pd-C dehydrogenates V at 310° to cadalene (VII). LiAlH $_4$  in  $Et_2O$  converts I to acordiol (VIII), amorphous, which is dehydrated with  $\alpha$ - $C_6H_5(CO)_2O$ , to  $C_{15}H_{16}$  (IX), b.p. 117-21°, and has 2 double bonds. IX with PtO $_2H$  yields VI and Pd-C dehydrogenates IX to VII. The cadalene skeleton probably is obtained by rearrangement. I on NaOH fusion at 225° yields piperitone and a doubly-unsatd. acid,  $C_{15}H_{18}O_2$  (X) (benzylthiogallium salt, m. 120°). No ester, b.p.

130°). X with PtO<sub>2</sub>-H yields a tetrahydro deriv., b, 180-2°. I with MeMgI yields methylacorolone (XII), m. 139°, which forms with LiAlH<sub>4</sub> methylacordiol (XIII), m. 119°. XIII dehydrates to a doubly unsatd. C<sub>10</sub>H<sub>16</sub> (XIV), b, 132°. XIII with Pd-C dehydrogenates to 7-methyl-cadalene, m. 39.5°, and minor amts. of azulene. I appears to show an anomalous CHI<sub>2</sub> reaction because an Ac grouping is not proved for I, and I dioxine subjected to a Beckmann rearrangement does not form volatile acids or amines. The data indicate for I the formula Ia, where the



5-position is favored for the 2nd CO group. I with BaH forms monobenzylideneacorone, C<sub>10</sub>H<sub>12</sub>O<sub>2</sub>, m. 185-6°. The results indicate position 7 for the reactive carbonyl while the 2nd carbonyl, probably in position 5, is sterically hindered. The failure of the characteristic FeCl<sub>3</sub> test for a  $\beta$ -diketone and ultraviolet spectra exclude  $\alpha$ - and  $\beta$ -diketone structures. The differences in the infrared spectra of acorane and tetrahydrocadinene suggest that I and II do not have the cadalene skeleton but form cadalene by rearrangement during dehydrogenation. N. T. F.

*CH**✓7*

**Neutral components of cork wax.** F. Šem and V. Balant (Tech. Univ., Prague). *Collection Czechoslov. Chem. Commun.*, **15**, 73-81 (1950) (in English). —The neutral components of cork wax were reinvestigated. Cork prep'd. from the bark of *Quercus rubra* averages 20% neutral components by cold extn. with EtOH-C<sub>6</sub>H<sub>6</sub>. Chromatographic sepn. produces a mixt. of friedelin, cerin, and C<sub>18</sub>H<sub>30</sub>O (I), paraffinic alcs., lactates m. 346°, benzoates m. 48-50°, *p*-nitrobenzoates m. 130-162°. I is probably a mixt. of C<sub>18</sub> to C<sub>24</sub> compds. CrO<sub>3</sub>-AcOH oxidizes I to C<sub>18</sub>H<sub>30</sub>O<sub>2</sub> (II) acid m. 75°. A tricyclic C<sub>24</sub>H<sub>30</sub>O (III) satis. alc. and a C<sub>24</sub>H<sub>30</sub>O<sub>2</sub> (IV), triterpene diol, and a C<sub>24</sub>H<sub>30</sub>O<sub>3</sub> (V) triterpene hydroxy ketone were found in succeeding fractions. A diterpene fraction which may be C<sub>20</sub>H<sub>30</sub>O or C<sub>20</sub>H<sub>30</sub>O<sub>2</sub> was isolated which m. 200-2° and had an approx. mol. wt. of 317. N. T. Farmaci

CA

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**Some aminoalkyl derivatives of benzimidazole.** F. Sorm and J. Urban (Tech. Univ., Prague). *Collection Czechoslov. Chem. Commun.*, 15, 109-209 (1950) (in English); cf. *C.A.*, 44, 1838. 2-(2-Aminoethyl)benzimidazole (**I**) is prep'd. in 3 ways: a)  $\text{C}_2\text{H}_5\text{NH}_2$ ; (**II**) heated 1.5 hrs. at 145° with double its wt. of  $\text{B}_2\text{NHC}_2\text{H}_5\text{CH}_2\text{CO}_2\text{H}$  gives 76% 2-(2-benzimidazolyl-benzimidazole) (**III**), converted by  $\text{EtOH-HCl}$  to the mono-*HCl salt*, m. 229-30°. **III** boiled 3 hrs. with 15% HCl gives 67% 1*H*-**IV**, softens at 280°, m. 325°. A soln. of 4.5 g. **II** and 4.9 g.  $\beta$ -alanine in 40 cc. 15% HCl is evap'd. and the residue heated 2 hrs. at 160° and ext'd. with MeOH, giving 45% 1*H*-**IV**, converted by  $\text{NH}_2\text{OH}-\text{CH}_2\text{Cl}$  to **I**, m. 160°. **I** rapidly absorbs  $\text{CO}_2$  from the air. Et-2-benzimidazole-propanoate heated with  $\text{NaBH}_4\text{-H}_2\text{O}$  in EtOH gives 87% of the *hydrazide*, decomp. 250° (from MeOH); this with iso-AmNH<sub>2</sub> in EtOH and HCl, followed by refluxing, gives 65% 2-[2-(2-*tert*-butylamino)ethyl]benzimidazole-*HCl*, decomp. 215-7°, which, refluxed 7 hrs. with concd. HCl, yields 93% 1*H*-**IV**. 2-Phenylbenzimidazole (**IV**) (9 g.) in 150 cc. hot dioxane treated with 7.5 g. 2-(1-piperidyl)ethyl chloride and 3 g. NaNH<sub>2</sub> and the mixt. re-

fluxed 6 hrs. gives, after filtration and solvent removal, 12.4 g. of a viscous oil, purified by crystn. and distn. to yield 1-[2-(1-piperidyl)ethyl]-2-phenylbenzimidazole (**V**), m. 144-7°; *tartrate*, m. 151°; *HCl salt*, m. 229-31°; *succinate*, **V**.  $\text{CaH}_2\text{O}_2$ , m. 120-1°. In the same way, 1.5 g. **IV**, 2.9 g.  $\text{Me}_2\text{NCH}_2\text{CH}_2\text{Cl}$ , and 1.5 g. NaNH<sub>2</sub> give 4.3 g. crude product that after sublimation yields crystals of 1*H*-2-(dimethylaminoethyl)-2-phenylbenzimidazole, m. 79-80.5° (forms a carbonate in air); *tartrate*, m. 191-4°; *succinate*, m. 135°. In the same way 4.2 g. 2-(*p*-methoxyphenyl)-benzimidazole, 3 g. 2-(1-piperidyl)ethyl chloride, and 1.2 g. NaNH<sub>2</sub> give 1-[2-(1-piperidyl)ethyl]-2-(*p*-methoxyphenyl)-benzimidazole, m. 101° (forms a carbonate in air); *HCl salt*, m. 218°.

David Todd

CA

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Proteins and amino acids. VI. A synthesis of proline and hygric acid. F. Šorm and J. Šmet (Central Chem. Research Inst., Prague). "Collection Czech Chem. Commun." 16, 42-6 (1951); cf. C.A. 45, 9482c. Tetrahydrofuran was converted into 4-acetoxy-1-bromobutane with AcHr then to 1-bromo-4-chloroaleric acid (I) through 4-acetoxycaprononitrile and 4-chlorovaleric acid. I was cyclized with 40% aq. NH<sub>3</sub> at 10° to m-proline in a 30% yield and isolated through the Cu salt, m. 105-8°, m-proline rhodanilate, m. 133 1° decompr. m-Hygric acid was obtained from I with 40% MeNH<sub>2</sub>, purified by sublimation at 140° at 1 mm. and recrystn. from CHCl<sub>3</sub>, m. 100-70°. Et m-hygrate by KI 4° was not resolved with d-bromocamphorsulfonic acid.

W. M. Potts

RECEIVED AND FILED  
JULY 1968 BY C. J. MARESH

The preparation of trinitrophloroglucinol. Fr. Šurc and C. Z. Drápalová, *Chem. Obzor* 12, 153-6 (1937). Ten g. of 1,3,5-trichloro-2,4,6-trinitrobenzene (I) and 15 g. of NaOH in 600 cc. of 85-90% EtOH were boiled for 1 hr. until flakes of Na trinitrophloroglucinate (II) formed in the soln. They were collected on a filter paper, washed with 50% EtOH and with concd. H<sub>2</sub>O<sub>2</sub>, and were dried at 100°. The original filtrate was treated with 5 cc. of H<sub>2</sub>O contg. 5 g. of NaOH and with 10 g. of I. A repetition of the procedure improves the yield of II. The total yield of II was 11 g. (55% of the theoretical yield). Beginning with 1,3,5-trinitro-2,4,6-triaminobenzene and boiling it in NaOH soln., the authors prepd. II in 70% yield. They prepd. and describe the Na, K, NH<sub>4</sub>, Li, Ca, Ba, Sr, Cd, Pd and Ag salts of II. Only the Pb and the Ag salts had properties of interest to makers of explosives.

Frank Mareš

## AIA-11A METALLURGICAL LITERATURE CLASSIFICATION

160000 4/2

161003 MAP ONE GEL

161004

MAP ONE GEL

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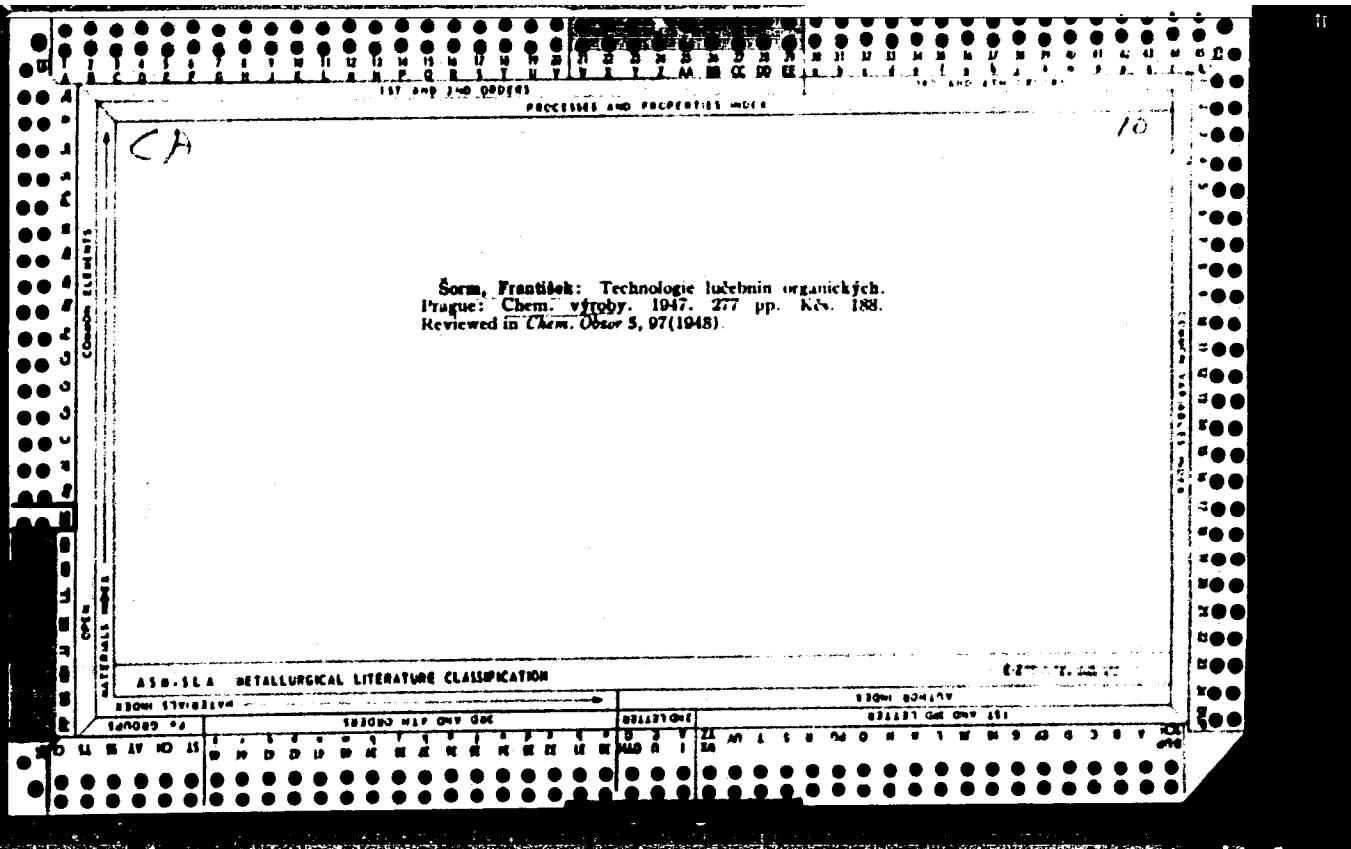
APPROVED FOR RELEASE: 08/25/2000

CIA-RDP86-00513R001652420013-7"

## ASA 554 - METALLURGICAL LITERATURE

The preparation of tetracyano-1,4-benzoquinone. I ran.  
[J. Am. Chem. Soc. 114, 39-40 (1992).] Tetrahydro-  
furan (1.0 g.), ground finely, screened through 1 mm. mesh,  
and suspended in 30 cc. EtOH, was mixed with 5.8 g. NaNO<sub>2</sub>  
for 8 hrs. in a rotating vessel. The yellow chloranide  
came converted into the red chloranidebenzoquinone  
which formed black-blue lustrous crystals of tetracyano-  
benzoquinone (I), forming a red-violet soln. in EtOH,  
Et<sub>2</sub>O and Et<sub>2</sub>CO, a blue soln. in CHCl<sub>3</sub> and toluene, and  
insol. in H<sub>2</sub>O. Because the oxidation of I in soln. was  
faster than the titration, it was not possible to titrate I  
titrimetrically. In an I-decomp., the crystals lost their  
luster, disintegrated, and left a yellow-green powder;  
the decomp. progressed as a linear function of the time,  
evolving elementary N (30% of the N in the mol. in 90  
days). I in acetone mixed with aq. KI adsorbed with dil.  
HCl, and稀. with water yielded a white ppt. of tetra-  
cyanoquinonimine (II). During the reaction it was no  
necessary to add acetone to the mixt., and to remove the ppt.  
I with a satd. Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> soln. The II, washed in EtOH and  
dried in a stream of CO<sub>2</sub>, was sol. in acetone, partly sol. in  
Et<sub>2</sub>CO, but insol. in H<sub>2</sub>O, in the air, particularly if moist, II  
oxidized to the blue quinone. In a flame I exploded  
violently, even in small quantities, whereas II merely  
ignited; the acceleration of the disruption is ascribed to  
the quinonoid structure of the C<sub>6</sub>H<sub>4</sub> nucleus. Although I had  
good detonating properties which it did not lose at high  
pressures (1000 kg. sq. cm.) it exploded at 0.1" and  
showed a sensitivity of 20 cm. in a 0.5 kg. Kast hammer;  
the gradual disintegration prevents its technical applica-  
tion, and among explosives I remains only of theoretical  
interest.

Frank Matesh



CA

## PROCESSES AND PROPERTIES INDEX

*Synthesis of homohygrinic acid.* F. Šorm (Soc. Chem. Met. Production, Prague-Vysočany). *Coll. Czech. Chem. Commun.*, 12, 375-80 (1947). —A new synthesis of homohygrinic acid (I),  $\text{MeN}(\text{CH}_2\text{CH}_2\text{CH}_2)\text{CH}_2\text{CO}_2\text{H}$ , gives

a product m. 124° differing from the I (m. 91°) isolated by Hess (cf. H. and Flak, *C.A.* 14, 3249) from the oxidation of the alkaloid cyclohygrine.  $\text{RCO}_2\text{H}$  ( $\text{R} = \text{tetrahydrofurylmethyl}$ ; prepd. by the steps  $\text{ROH} \rightarrow \text{RBr} \rightarrow \text{RCN} \rightarrow \text{RCO}_2\text{H}$ ) (11.5 g.), added to an ice-cooled mixt. of 90 ml. 48% aq.  $\text{HBr}$  and 450 ml.  $\text{Ac}_2\text{O}$ , heated 5 hrs. under pressure to 100°, the volatile matter distd. off, and the residue taken up in ether, washed, and dried, yielded 24.3 g. of an oil,  $\beta,\beta$ -dibromocaprylic acid (II); amide m. 72° (from  $\text{CdCl}_2$ ). II (56 g.) and 240 ml. 20%  $\text{MeNH}_2$  in  $\text{MeOH}$  were heated in 5 sealed tubes 6 hrs. at 110-30°, the volatile matter distd. off, the residue dissolved in 1 l.  $\text{H}_2\text{O}$ , and 240 g.  $\text{Ba(OH)}_2$  added. After standing 48 hrs., carbonation, acidification with  $\text{H}_2\text{SO}_4$ , filtration, and evapn. gave crude I, which did not crystallize. Distn. followed by treatment with  $\text{HCl}$  in abs. alc. gave 16.6 g. of the Et ester (III), b.p. 97-9°, a sweetish-smelling oil; picrate, yellow needles from alc., m. 112°. Hydrolysis of III by  $\text{Ba(OH)}_2$  gave I (isolated as before) which rapidly crystd. in a desiccator, m. 124° (from  $\text{CdCl}_2$ ); the  $N,N$ -dimethylamide (IV) b.p. 137-40° (picrate, needles, m. 147° (from alc.)).

Richard G. Kadesch

## ADM-11A METALLURGICAL LITERATURE CLASSIFICATION

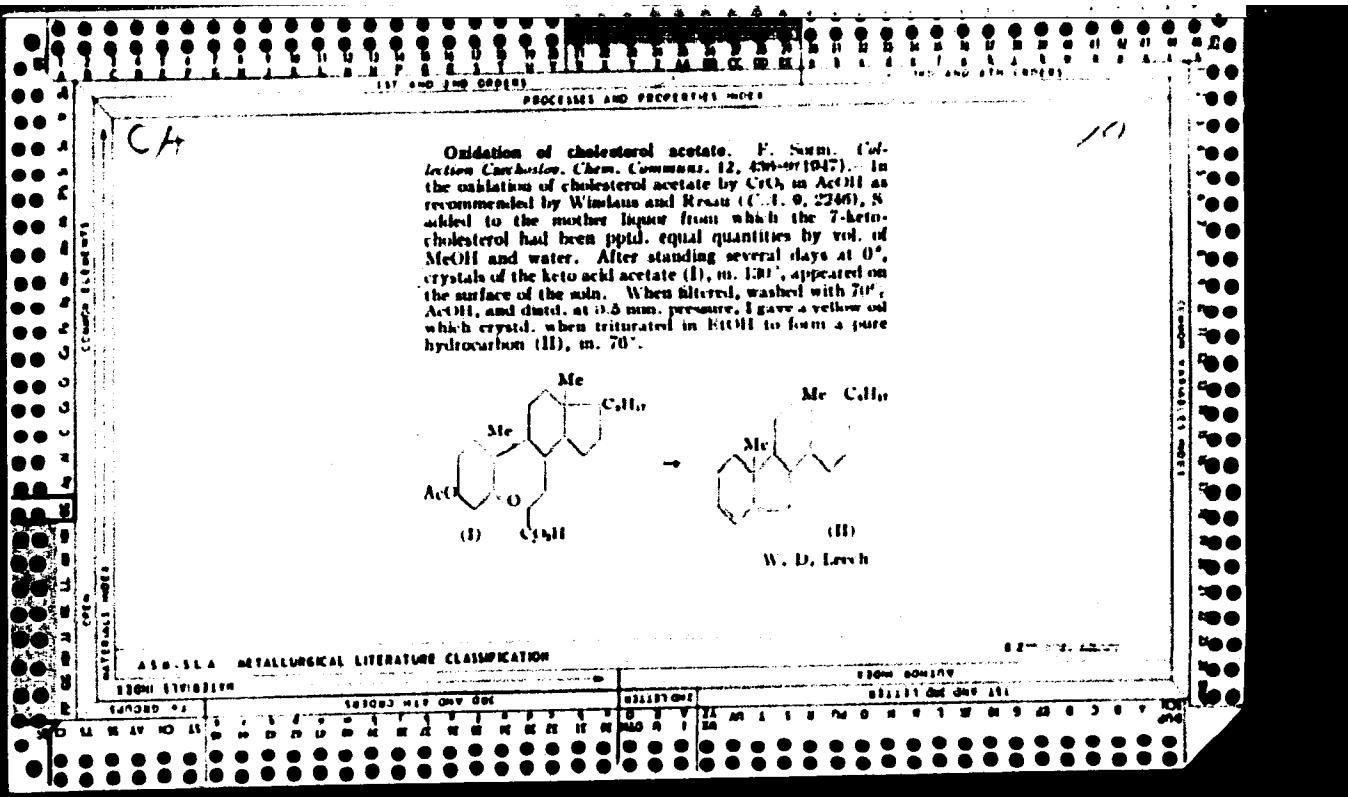
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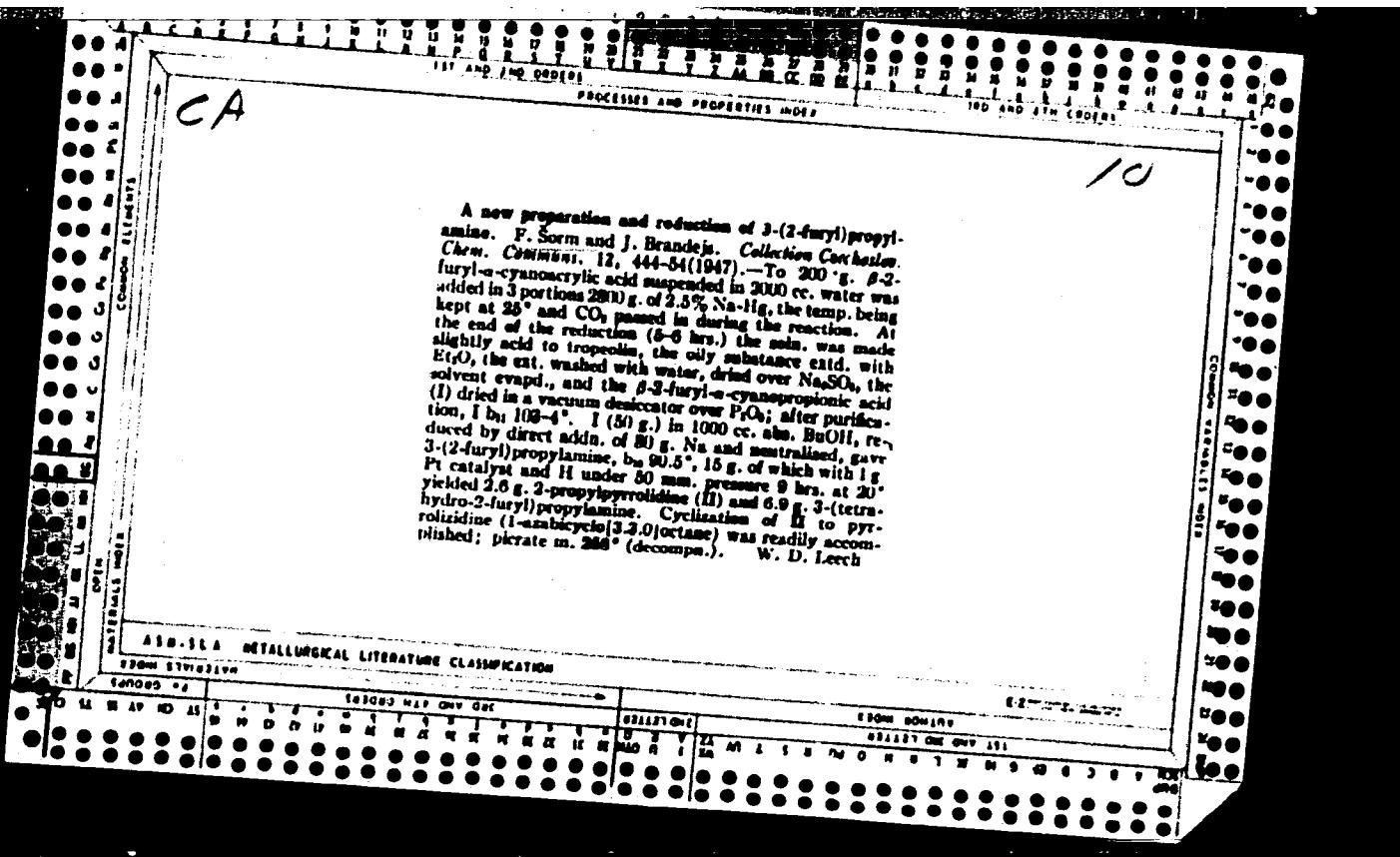
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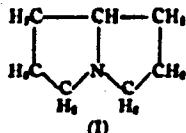
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A new synthesis of pyrrolidine (1-azabicyclo[3.3.0]-octane). F. Sorm and Z. Arnold. *Collection Czechoslov. Chem. Commun.* 12, 467-71 (1947).—To 100 g. of 48% HBr was added dropwise at 0° 320 cc. Ac<sub>2</sub>O, then 6 g. 3-(tetrahydro-2-furyl)propylamine, the mixt. heated on a water bath 6 hrs., the acids removed by vacuum distn., and the solid residue recrystd. from AcOEt, giving



4,7-dibromo-1-aminobutane-HBr, m. 117°; this was condensed directly to the 2-ring compd. by heating in NaOH soln. at 40° C. 2 hrs., from which the pyrrolidine (I), b. 140° (picrate, m. 258°), was isolated. I was also prep'd. by catalytic dehydration of 3-(2-furyl)-propylamine over Al<sub>2</sub>O<sub>3</sub> at 400° to the trimethylenepyrrole, which was transformed into I by catalytic reduction with Pt in anhyd. AcOH. W. D. Leech

## ABD-3 LA METALLURGICAL LITERATURE CLASSIFICATION

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SCAR, F.

**Sesquiterpenes. III. Synthesis of 4,6-dimethylazulene.** E. Sorm, Z. Sormová, and L. Šalivý. *Collection Czechoslov. Chem. Commun.*, 12, 551-61 (1947). Et 2-oxo-*tert*-cyclopentanecarboxylate heated 2 hrs. in the presence of Na in PhMe with  $\text{MeCHBrCO}_2\text{Et}$  gave, on distn., 85% Et 1-(1-carbethoxyethyl)-2-oxocyclopentanecarboxylate (I), b.p. 110°. I heated with concd. HCl 12 hrs. yielded 52%  $\alpha$ -methyl-2-oxocyclopentanecarboxylic acid, m.p. 151°; semicarbazone, m.p. 188°. Et ester (III), from II in abs. EtOH satd. with gaseous HCl, b.p. 81-3°. In the presence of K, abs. EtOH, and  $\text{NCCH}_2\text{CO}_2\text{Et}$ , III yielded 35% Et 2-(1-carbethoxyethyl)- $\alpha$ -cyano- $\Delta^5$ -cyclopentanecarboxylate (IV), b.p. 150-60°, reduced by hydrogenation in EtOH in the presence of PtO<sub>2</sub> to 82% Et 2-(1-carbethoxyethyl)- $\alpha$ -cyano-*cis*-cyclopentanecarboxylate, b.p. 151°. When 3 g. IV was boiled 10 hrs. with 30 cc. concd. HCl, there appeared on the reaction surface an oil which was sep'd. by steam distn., giving a total of 0.5 g. 2-methylbicyclo[3.3.0]octan-3-one (V); semicarbazone, m.p. 201°. The aq. layer yielded *cis*- $\alpha$ -methyl-1,2-cyclopentanedi- $\alpha$ -carboxylic acid (Va), m.p. 189°. From Et 2-carbethoxy-

methyle)- $\alpha$ -cyano-*cis*-cyclopentanecarboxylate, Na, C<sub>6</sub>H<sub>6</sub>, and MeI was obtained 81% Et 2-(carbethoxymethyl)- $\alpha$ -cyano- $\alpha$ -methylcyclopentanecarboxylate (VI), b.p. 157-9°, 40 g. of which heated with 500 ml. concd. HCl 20 hrs. gave, on cooling, 18 g. pure *cis*-acid (Va), m.p. 181°; from the mother liquors was obtained 2 g. *trans*-acid (Vb), m.p. 150°. Va and  $\text{SOCl}_2$  gave the acid chloride, converted by  $\text{CH}_2\text{N}_2$  through the diamide into *cis*- $\beta$ -methyl-1,2-cyclopentanedipropionic acid (VII), m.p. 151°. From VII, reduced Fe powder, and  $\text{Ba(OH)}_2$  was distd. 83% 2-methylbicyclo[3.3.0]octan-4-one (VIII), b.p. 123°; semicarbazone, m.p. 207-8°. Hydrolysis of the reaction product of VIII and  $\text{MeMgI}$  yielded 94% *cis*-2,4-dimethylbicyclo[3.3.0]octan-4-ol, b.p. 135-0°; dehydrated over  $\text{KHSO}_4$  to 88% *cis*-2,4-dimethylbicyclo[3.3.0]-3(*ox* 4)-decene, b.p. 198.5°; dehydrogenation of the latter over 10% Pd charcoal at 310-60° yielded crude 4,6-dimethylazulene (IX), which could not be crystd.; with trinitrobenzene, dark needles (X), m.p. 113°, were obtained. IX, liberated from X chromatographically in pentane soln., was light purple in color. M. Q. Webb

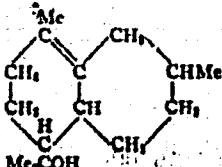
CA	PROCESS AND PROPERTY INDEX													
<p>The effect of the Reformatskii reagent on <i>N</i>-methyl imides of dicarboxylic acids. II. Effect of organometallic reagents on the amide group. J. R. Lukeš and F. Šorm (United Chem. Met. Manuf., Prague-Vysokany). Collection Czechoslov. Chem. Commun. 12, 637-40 (1947) (in French); cf. C.A. 34, 7008. — <i>N</i>-Methyl-phthalimide (I) (63 g.), 165 g. BrCH<sub>2</sub>CO<sub>2</sub>Rt (II), and 150 cc. C<sub>6</sub>H<sub>6</sub> were heated with a few Zn turnings in an oil bath at 100–10°; after an initial violent reaction 80 g. Zn was added over 2 hrs. and heating continued 10 hrs. Addn. of dil. HOAc, extn. with C<sub>6</sub>H<sub>6</sub>, and distn. of the combined C<sub>6</sub>H<sub>6</sub> exts. gave 20 g. unconverted I, b.p. 122°, and 6 g. <i>Ei</i> 1-methyl-2-oxo-1,2,3,4-tetrahydro-5-pyruvatoate, b.p. 103°, crystal. in contact with H<sub>2</sub>O, m. 62°. Similarly <i>Ei</i> 4-methyl-1-oxo-2,3-dihydro-1,4-dihydro-5-acetate (or <i>Ei</i> 4-methyl-1-oxo-3,4-dihydro-1,4,2<i>H</i>-oxazine-5-acetate), obtained in 6-g. yield from 60 g. <i>N</i>-methylphthalimide (III) (m. 76°), 150 cc. C<sub>6</sub>H<sub>6</sub>, 165 g. II, and 80 g. Zn, b.p. 161°; 20 g. unreacted III was obtained, b.p. 110–30°. <i>Ei</i> 2-methyl-1-oxo-3-hydroxy-3-isovalinoacetate (IV), prep'd. in 15-g. yield by treating 40 g. <i>N</i>-methylphthalimide with 180 cc. C<sub>6</sub>H<sub>6</sub>, and 85 g. II at 110°, adding 40 g. Zn, then heating at 120° 5 hrs., m. 106° (3 recrystns. from EtOH). IV is stable; it can be heated to the b.p. without decompr. <i>N</i>-Methylphthalimide reacted similarly as above, and a high-boiling fraction was obtained but no chem. compd. The various <i>N</i>-methyl imides were prep'd. by distn. of the dicarboxylic acids with MeNH<sub>2</sub> at 700 mm. and crystn. from C<sub>6</sub>H<sub>6</sub> or C<sub>6</sub>H<sub>6</sub>-ligroin. Low yields of ester products may be attributed to appreciable H<sub>2</sub>O solv. J. W. G.</p>														
ASB-LSA METALLURGICAL LITERATURE CLASSIFICATION														
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U.S. A. V. NO. 15	P	H	D	P	K	M	H	V						

SORM, F.

CA

Terpenes. V. The terpenic constituents of the essential oil of sweetflag (*Acorus calamus L.*). F. Sorm and V. Herout. *Collection Czechoslov. Chem. Commun.* 13, 777-200 (1948); cf. C.A. 42, 7283b.—Oil of Dutch origin was subjected to fractionation after sepn. of acidic and phenolic substances. The lower-boiling fraction, b.p. 42-70°, contained terpenes which polymerized upon standing and were not further examd. *d*-Camphor was isolated as was a terpenic alc.,  $C_{10}H_{16}O$  (semicarbazone m. 133°), and an alc. ( $C_{10}H_{16}O$ ) fraction which was unsatd. (1 double bond/mol.). From these substances arose the characteristic odor of this essential oil. Calamene,  $C_{10}H_{16}$ , d<sub>4</sub> 0.9226, n<sub>D</sub> 1.5040, was found to be a mixture of a bicyclic hydrocarbon (2 double bonds) and a tricyclic hydrocarbon (1 double bond). A fraction ( $C_{10}H_{16}O$ ), probably possessing 2 double bonds and a carbonylic

O and named acoroxide, was easily hydrogenated to  $C_{10}H_{18}O$ , which contained no active H and did not form a semicarbazone or react with PrMgBr. A ketone, calamone,  $C_{10}H_{16}O$ , b.p. 103-0°, was converted to the semicarbazone, m. 185-7°. There were also isolated 2 isomeric diketones,  $C_{10}H_{16}O_2$ , acorone, m. 100-1°, and iso-acorone, m. 103-7°, and a substance,  $C_{10}H_{16}O$ , m. 103°, called calamone. VI. Constitution of carotol. II. F. Sorm and L'Urbauk. *Ibid.* 420-7.—A series of reactions was carried out on carotol which indicated that its structure is



M. O. Webb

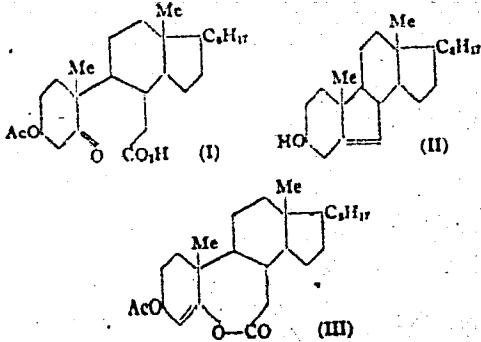
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Sorm, F.

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**PROCESSES AND PROPERTIES INDEX**

**$\beta$ -Norcholesterol.** F. Sorm and H. Dykova. *Collection Czechoslov. Chem. Commun.*, 3, 407-19 (1948) (in English).—The principal reaction product (I) from the  $\text{Cr}_2\text{O}_7$ -oxidation of cholesterol acetate (*Collection Czechoslov. Chem. Commun.*, 12, 437 (1947)), was used for the prepn. of  $\beta$ -norcholesterol (II), an analog of cholesterol with a 6-membered B ring. A white needlelike enol lactone (III) with a 7-membered ring, m. 122° (from MeOH),  $[\alpha]_D^{25} - 60^\circ$  (c 2,  $\text{CHCl}_3$ ), was obtained in either 1-g. yield (52%)



after refluxing 2 g. I in 30 cc.  $\text{Ac}_2\text{O}$  on a water bath 1 hr., removing  $\text{Ac}_2\text{O}$ , sepr. the noncryst. residue on  $\text{Al}_2\text{O}_3$  by chromatography, and recrystg. the petr. ether eluate from  $\text{MeOH}$ , or in 6 g. yield (63.4%) by treating 10 g. I in 20 cc. dry pyridine with 8.8 g.  $\text{BzCl}$  at room temp. 72 hrs. and purifying the  $\text{Et}_2\text{O}$  ext. of the reaction mixt. III (4 g.) heated in a test tube on an oil bath 30 min. at 180-200° evolved  $\text{CO}_2$  and formed 01.4%  $\beta$ -norcholesterol acetate (IV), m. 78° (from  $\text{Me}_2\text{CO}$ ),  $[\alpha]_D^{25} - 59^\circ$  (c 2,  $\text{CHCl}_3$ ). II, fine white needles from abs.  $\text{MeOH}$ ,  $\text{EtOH}$ , or petr. ether or a voluminous powder from aq.  $\text{EtOH}$ , m. 114°,  $[\alpha]_D^{25} - 90^\circ$ , was obtained in 96.3% yield by refluxing 3.00 g. IV in 800 cc. boiling  $\text{MeOH}$  with a satd. aq. soln. of KOH (5 g.) on a water bath 2 hrs. and purifying the product by removing the  $\text{MeOH}$ , taking up the residue in  $\text{Et}_2\text{O}$ , washing it until neutral, drying it with  $\text{Na}_2\text{SO}_4$ , distg. off the  $\text{Et}_2\text{O}$ , and recrystg. II was identified as its benzate (V) (200 mg.), fine white crystals, m. 136° (from  $\text{EtOH}$  and  $\text{Me}_2\text{CO}$ ),  $[\alpha]_D^{25} - 64^\circ$  (c 4,  $\text{CHCl}_3$ ), resulting from the reaction of 200 mg. of II in dry pyridine with  $\text{BzCl}$  at room temp. for 72 hrs., and as its *sulfuric acid ester* (VI), white crystals from petr. ether, m. 108°,  $[\alpha]_D^{25} - 52.8^\circ$  (c 3.33,  $\text{CHCl}_3$ ), resulting from the reaction of 500 mg. II with 5 times the theoretical amt. (775 mg.) of  $\text{SOCl}_2$  at room temp. for 30 min., followed by removal of the excess  $\text{SOCl}_2$  with  $\text{H}_2\text{O}$  and purification of the product. II (1 g.) in 10 cc. dry  $\text{C}_6\text{H}_6$  and 7.6 cc. dry  $\text{Me}_2\text{CO}$  was con-

**ASH-SLA METALLURGICAL LITERATURE CLASSIFICATION****MATERIALS INDEX****SUBJECT INDEX****APPENDIX INDEX**

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SORM, F.

Terpenes. IV. Constitution of carotol. F. Sorm and  
L. Urbanek. Collection Czechoslov. Chem. Commun. 13,  
pp. 57-61 (1948). Hydrogenation of the sesquiterpene alc.  
carotol,  $[\alpha]_D^{25} -6.9^{\circ}$ ,  $n_D^{20} 1.4997$ ,  $d_4^{20} 0.9702$ , obtained  
from the oil of *Daucus carota*, gave dihydrocarotol (I),  
colorless oil after distn.,  $b.p. 131-3^{\circ}$ . On the basis of  
the oxidation of carotol with  $K_2MnO_4$  and other reactions,  
S. and U. suggest that carotol contains 1 double bond  
in the hydrogenated skeleton of 1,7-dimethyl-1,3,5-trisopropyl-  
1-ophthalidene. T. B. N.

CA

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Reduction of pyridinecarboxylic acids. F. Sorm.  
Collection Czechoslov. Chem. Commun. 13, 57-73 (1948).  
The reduction of the 3-pyridinemono-carboxylic acids was  
carried out by each of the following methods: catalytically,  
by Zn and AcOH, and electrolytically. By catalytic re-  
duction piperidine acid, m.p. 202°, was obtained from pic-  
oline acid. On catalytic reduction isopropyl picoline acid pro-  
duced isopropionic acid and piperidine. With Zn and  
AcOH, picoline acid produces 2-piperidine and isopropionic  
acid produces 4-piperidine Thomas H. Niven

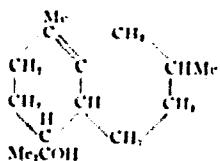
A4-11A METALLURGICAL LITERATURE CLASSIFICATION

PROCESSES AND PROPERTIES INDEX  
1ST AND 2ND ORDERS

CA

Terpenes. V. The terpene constituents of the essential oil of sweetflag (*Acorus calamus* L.). F. Sorm and V. Herout. *Collection Carlsbad. Chem. Commun.* 19, 177-213 (1948); cf. C.A. 42, 7283b.—Oil of Dutch origin was subjected to fractionation after sepn. of acidic and phenolic substances. The lower-boiling fraction, b.p. 42-70°, contained terpenes which polymerized upon standing and were not further examined. Camphor was isolated as was a terpene alc.,  $C_{10}H_{16}O$  (semicarbazone m.p. 183°), and an alc. ( $C_{10}H_{16}O$ ) fraction which was unsatd. (1 double bond/mol). From these substances arose the characteristic odor of this essential oil. Calamene,  $C_{10}H_{16}$ , d<sub>4</sub> 0.9280, n<sub>D</sub><sup>20</sup> 1.5440, was found to be a mixture of a bicyclic hydrocarbon (2 double bonds) and a tricyclic hydrocarbon (1 double bond). A fraction ( $C_{10}H_{16}O$ ), probably possessing 2 double bonds and a carbonyl

O and named acacoxide, was easily hydrogenated to  $C_{10}H_{16}$ , which contained no active H and did not form a semicarbazone or react with PhMgBr. A ketone, calamone,  $C_{10}H_{16}O$ , b.p. 90-91°, was converted to the semicarbazone, m.p. 183.7°. There were also isolated 2 isomeric diketones,  $C_{10}H_{12}$ , acetone, m.p. 140.1°, and its isomer, m.p. 93.7°, and a substance,  $C_{10}H_{16}O$ , m.p. 168°, called calamone. VI. Constitution of carotol II. F. Sorm and L. Uhlík. *Biol.* 15, 7. A series of reactions was carried out on carotol which indicated that its structure is



M. Q. Webb

ASH-SLA METALLURGICAL LITERATURE CLASSIFICATION

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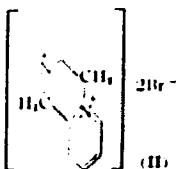
## PROCESSES AND PROPERTIES INDEX

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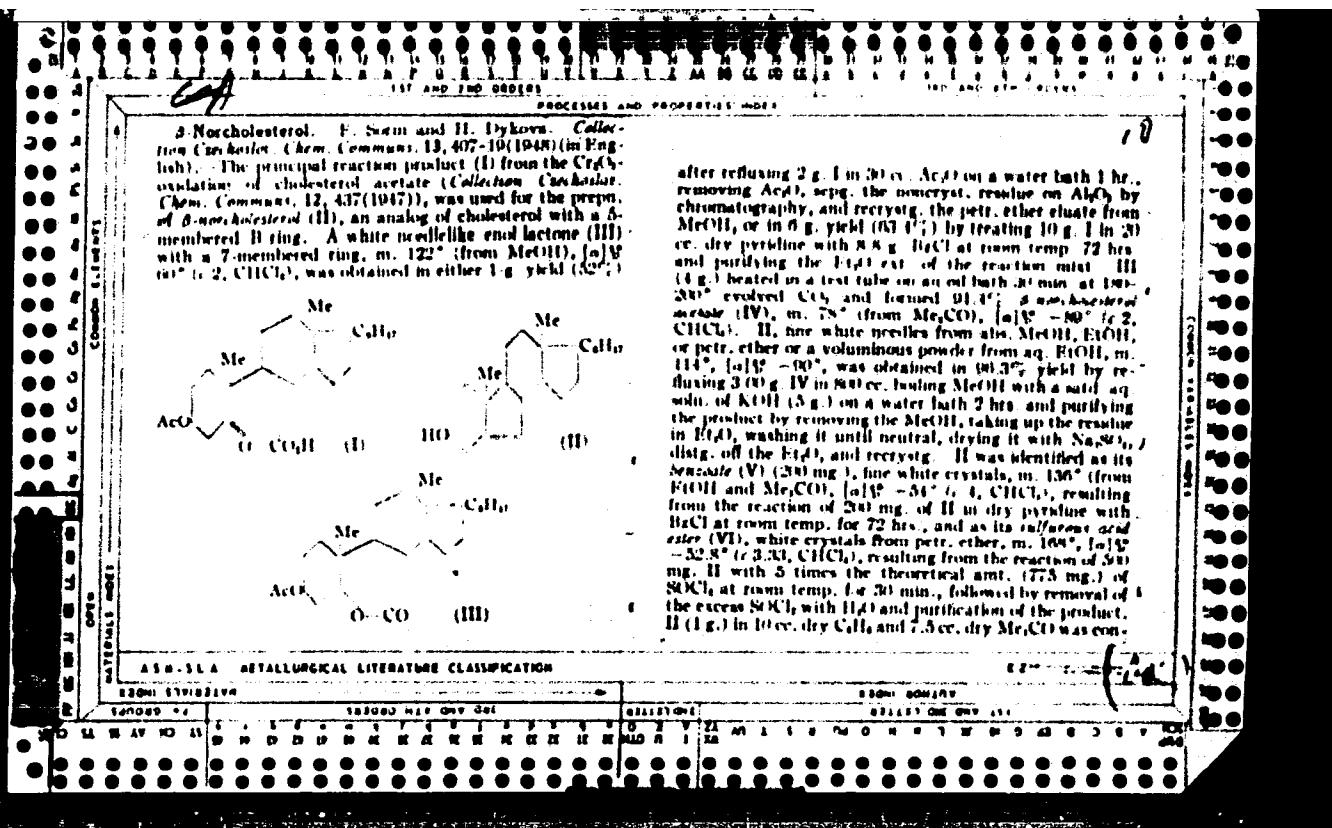
**Preparation of pyridine carbinols and some of their reactions.** P. Stein and L. Sedivý, *Collection Czechoslov. Chem. Commun.*, **13**, 289-99 (1948) (in English); *Chem. Abstr.* **40**, 175 (1945).—By reduction of picolinic and isonicotinic acids with Zn and AcOH 2-pyridinemethanol,  $b_p$  112°, and 3-pyridinemethanol,  $b_p$  154°, were prep'd. 2-(Bromomethyl)pyridine-HBr (I) was prep'd. by adding 68 ml. of 48% HBr drop by drop to 250 ml. ice-cooled  $\text{AgO}$  with const. stirring, then 6 g. 2-pyridinemethanol added, the soln heated in a glass autoclave 3 hrs. to 100° on a water bath, and the AcOH distd. off, together with HBr. Cryst. from EtOH yielded 1 as practically colorless needles, m. 140°. I (20.5 g.) in a small amt. of H<sub>2</sub>O was covered with 150 ml. benzene and mixed with 10 g. KOH in H<sub>2</sub>O; the (bromomethyl)pyridine srgng. as an oil was taken up in the benzene, the benzene layer dried with anhyd. Na<sub>2</sub>SO<sub>4</sub>, and refluxed on a water bath for 100 hrs.; the bis(methylenebispyridinium bromide) (II) which sep'd. during the heating crystd. from

contg. 2 moles (I) of excess. II hydrogenated with H and a PtO<sub>2</sub> catalyst produced the HBr salt of di-1-piperidyl-piperazine, the base being liberated from a const. of soln. of the salt with KOH. The oily base taken up in EtOH, dried over KOH, and distilled, b.p. 170°, the distillate crystd. at once and m. 81°. By refluxing 4-(bromomethyl)pyridine, a polymeric quaternary salt is obtained.

Thomas B. Niven



aq. MeOH has almost colorless needles, m. 200° (decompn.).



vered into  $\beta$ -*iso*-cholestene (VII) by the oxidation method of Oppenauer by treating it with six mg. of  $(\text{MeCO})_2\text{Al}$  in 5 cc. dry  $\text{CHCl}_3$  at  $75-80^\circ$  for 1 hr. isolated as its semicarbonate, m.  $251^\circ$ . The ultraviolet absorption of VII showed it was  $\alpha,\beta$  unsatd. Fppts. to definitely establish the structure of III, as well included the mode of formation and the structure of II. In addition, the structure of IV. The inability to titrate III as product of the absence of a  $\text{CO}_2\text{H}$  group in III as shown by potentiometric titration; a quant. recovery of the Me ester of I, m.  $79^\circ$ , from the reaction mixt. of 300 mg. of that substance and 200 mg.  $\text{HgCl}_2$  instead of recovery of III; and the failure of III to form an ester with  $\text{CH}_3\text{N}_2^+$  all indicated that the  $\text{CO}_2$  and  $\text{COH}$  groups of I were involved in the formation of III. The assumed enol lactone structure of III seemed reasonable since its ultraviolet absorption spectrum approached that of  $\text{CH}_3\text{CHOAc}$  and not of  $\text{CH}_3\text{CHCO}_2\text{H}$ , even though its splitting off of  $\text{CO}_2$  to form IV was unusual. Analysis of IV showed the  $\text{AcO}$  group in

ring A was maintained; and the neg. rotation of IV, in agreement with the characteristic rotational changes in cholesterol-type compounds, as opposed to the pos. rotation of I and III, showed ring B was closed. Four lines of evidence are given to show the presence of only 1 double bond in IV: (1) absorption of 0.18 cc. II, 0.26 cc., theoretical) at  $0^\circ$ ; 700 mm., to form  $\text{diisopropyl iso-cholesterol acetate}$  (VIII) from 100.5 mg. IV added to prehydrogenated catalyst (100 mg.  $\text{PtCl}_2$  in glacial  $\text{HgOAc}$ ); (2) formation of  $41^\circ$  (2 g.)  $\beta$ -*iso*-cholesterol acetate anide (IX) (from  $\text{MeOH}$ ), m.  $108^\circ$ ; (3)  $\text{IV} - 34^\circ$  ( $2, \text{CHCl}_3$ ), by the reaction of 2.072 g. IV and 80 cc. 0.4 N ( $100^\circ$ ); (4) evn. per-  
titration of the reaction mixt. with 90.1 cc. 0.1 N  $\text{Na}_2\text{S}_2\text{O}_3$  (100 cc., theoretical); (5) absorption by IV of an amt. of  $\text{Hg}_2$  in  $\text{Et}_2\text{O}$  and glacial  $\text{HgOAc}$  corresponding to 1 double bond; and (4) formation of the cryst.  $\text{HgCl}$  addn. product of IV, m.  $80^\circ$  (mixed m.p. with IV,  $60^\circ$ ), by soln. of 2 cc.  $\text{CHCl}_3$  contg. 414 mg. IV with gaseous  $\text{HgCl}$  at  $0^\circ$ .

II-1. Whelden

CA

The synthesis of sparteine and isosparteine. P. Sorm and B. Keil. *Collection Czechoslov. Chem. Commun.* 13, 544-551 (1948) (in English). — The by-product in the prepn. of di-Me  $\alpha,\gamma$ -di-2-pyridylglutarate (I) (C.A. 42, 6026b) was shown to be Me 4-oxo-3-(2-pyridyl)-1-pyridocoline-carboxylate (II) formed from I during distill. under reduced pressure by the simultaneous splitting off of MeOH and dehydrogenation. II, b.p. 270-5°, m. 169° (from *CaH*, petr. ether) [picrate, m. 239° (decomp.) (from diazome)], was obtained from Me 2-pyridineacetate (III) in 13% yield with  $\text{CH}_2\text{O}$ , and 25.5% yield with  $\text{CH}_3\text{O}$ . III (0.42 g.), 0.385 g.  $\text{HC}(\text{OEt})_2$ , and 0.5 cc.  $\text{Ac}_2\text{O}$  were heated 2 hrs. at 125-30°, the  $\text{EtOAc}$  and excess  $\text{Ac}_2\text{O}$  distilled, and the residue distilled, to give 2 fractions,  $b_p$  160° and  $b_p$  220° (IV), which crystallized. IV in *CaH* was chromatographed on 3 g. neutral  $\text{Al}_2\text{O}_3$ ; the first 10 cc. of *CaH* eluate gave yellow needles of II, m. 169°. The identity of the II obtained by the 2 methods was shown by mixed m.p. and identical absorption curves in the visible spectrum. I (7.8 g.) in 100 cc. glacial  $\text{AcOH}$  and 0.78 g. Adams PtO<sub>2</sub> at room temp. and 140 mm. Hg excess pressure of H absorbed 3333 cc. II and was worked up to give 0.8 g.,  $b_p$  98-100°, and 3.4 g.,  $b_p$  m. 173° (V). V (7 g.) in thiophene-free *CaH* was passed through 210 g.  $\text{Al}_2\text{O}_3$ . The following fractions of 200 cc. were collected and evapd. separately (fraction no., eluent, wt. in g.): 1. *CaH*, 0; 2-10, *CaH*, 1.21; 11-12, *CaH*, 0; 13-15,  $\text{CHCl}_3$ , 0; 16-20,  $\text{Et}_2\text{O}$ , 0, and 21-4,  $\text{EtOAc}$ , 1.49. The crystal. substance, m. 118-23°, from fractions 2-12 was sept. into VI and VII; fractions 21-4 yielded VIII. VI (0.36 g.) in 10 cc. 50%  $\text{H}_2\text{SO}_4$  was reduced electrolytically at 20-22° during 5.5 hrs. at 13 amp. with activated electrodes of pure Pb, made alk. with 15 g. solid  $\text{Ba}(\text{OH})_2$ , steam-distilled, and the steam distil-

I did not crystallize. Fractions 2-12 crystall. on wetting with  $\text{Et}_2\text{O}$  and on fractional cryst. from  $\text{Et}_2\text{O}$  gave 0.60 g. colorless prisms, m. 171° (VI), and 0.3 g. longish prisms, m. 181° (VII) (mixed m.p. showed a depression of 13°), while the motherliquor, mother liquor on further chromatography gave 0.53 g. VII. VI, m. 172° after 2 recryst. from  $\text{Et}_2\text{O}$  and sublimation *in vacuo*, was identical with the dioxosparteine previously described; VII, m. 135° after 2 recryst. from  $\text{Et}_2\text{O}$  and sublimation *in vacuo*, was shown to be another dioxosparteine isomer. Fractions 21-6 and 26-7 did not crystallize on wetting with  $\text{Et}_2\text{O}$ . Fractions 26-7 and 28-31 crystall. on wetting with *CaH* and were recryst. from  $\text{EtOAc}$ - $\text{Et}_2\text{O}$  to give Me 4-oxo-3-(2-piperidyl)octahydro-1-pyridocolinecarboxylate (VIII). II (3.8 g.) in 70 cc. glaced  $\text{AcOH}$  and 0.4 g. Adams PtO<sub>2</sub> gave as above 3.4 g. of a colorless resinlike substance which was heated 2 hrs. at 160-210° *in vacuo*. The residue, 2.9 g., was noncryst. and after soln. in 10 cc. *CaH* was chromatographed through 120 g. neutral  $\text{Al}_2\text{O}_3$ . Fractions of 100 cc. were taken and evapd. separately (fraction no., eluent, and wt. in g.): 1. *CaH*, 0; 2-10, *CaH*, 1.21; 11-12, *CaH*, 0; 13-15,  $\text{CHCl}_3$ , 0; 16-20,  $\text{Et}_2\text{O}$ , 0, and 21-4,  $\text{EtOAc}$ , 1.49. The crystal. substance, m. 118-23°, from fractions 2-12 was sept. into VI and VII; fractions 21-4 yielded VIII. VI (0.36 g.) in 10 cc. 50%  $\text{H}_2\text{SO}_4$  was reduced electrolytically at 20-22° during 5.5 hrs. at 13 amp. with activated electrodes of pure Pb, made alk. with 15 g. solid  $\text{Ba}(\text{OH})_2$ , steam-distilled, and the steam distil- (acca)

late made neutral to methyl orange and taken to dryness to give 0.24 g. HCl salt (IX). IX (0.21 g.) in 2 cc. hot H<sub>2</sub>O was added dropwise to 0.51 g. Na picrate in 10 cc. H<sub>2</sub>O, and the crude picrate which settled washed with abs. alc. and recrystd. from glc.-dioxane to give 0.44 g. of the picrate, m. 222° (decomp.)., presumably the dipicrate of



sparteine (X). VII on similar treatment gave 2 picrates, m. 187-9° (from alc.-dioxane) (XII) and m. 201° (from alc.-dioxane) (XIII), which were anal. mechanically. XII may be the dipicrate of  $\alpha$ -isoparteine while XIII appeared to be similar to X (cf. C.A. 28, 4084; 35, 30319). By examination of the structure of sparteine, if rings B and C are *cis* to each other, rings A and D can be attached to ring B or C, resp., either *cis* or *trans*. Thus, 3 isomersides can exist: (1) A:B *cis*, C:D *cis*; (2) A:B *cis*, C:D *trans*; and (3) A:B *trans*, C:D *trans*. Each of these isomersides will be resolvable into optical antipodes.

H. L. Yale

OA

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The electrolytic reduction of some keto acids. II  
R. Euker and P. Soren. *Collecting Cyclopedia Chem. Commun.*, 12, 485 (1948) (in English). Cf. C. I., 23, 1009. The following keto acids were subjected to reduction, electrolytically and by the method of Clemmensen:  $\gamma$ -acetylbutyric acid (I),  $\gamma$ -propionylbutyric acid (II),  $\gamma$ -diketocaprylic acid (III),  $\beta$ -acetoglutaramic acid (IV), hydrochloride (2-ketopimelic) acid (V), diethylene (VI), and 4-ketoazelaic acid (VII). Reduction to the parent acid proceeded smoothly in the majority of cases. Electrolytic reductions were accomplished by dissolving the keto acid in  $\text{HgSO}_4$  solution employing a d.c. of 5-10 amp and total of 1400 amp-hr. Cf. C. I., 6, 1929. I and II gave as sole products the corresponding normal acids in good yields. III gave the expected caprylic acid in very low yield only; the principal part of III underwent fission, with valeric acid being formed. Of the keto dicarboxylic acids the reduction proceeded smoothly with IV and V whereas VII resisted electrolytic reduction. On Clemmensen reduction V and VII show exactly the opposite behavior. The electrolytic reduction of VI proceeded very smoothly, giving very pure sebacic acid, and no fission, as with III, even though the 2 acids are constitutionally very similar.  
R. F. Dunbar

SORM, F.

C Z E C H

✓ Mechanism of antibiotic action. V. Effect of chloramphenicol, chlortetracycline, and oxytetracycline on the synthesis of glutamic acid decarboxylase in *Escherichia coli*, and of tyrosine decarboxylase in *Streptococcus faecalis*. D. Grünberger, J. Škoda, and F. Sorm (Česk. akad. věd, Prague). *Chem. Listy* 48, 13827d. — Chlortetracycline (Aureomycin) and oxytetracycline (Terramycin) as well as chloramphenicol, inhibit the formation of glutamic acid decarboxylase in *E. coli*. All 3 inhibit the growth of *Streptococcus faecalis*, but do not influence the formation of tyrosine decarboxylase. M. Hudlický.

CA

Terpenes. VII. A new colored hydrocarbon from the oil of wormwood. P. Šurma, F. Vondráček, and V. Herout. Collection Česk. Časopis. Chem., Commun., 14, 91-7 (1949) (in English); cf. C.A. 43, 3816e; 44, 615e.—Sepn. by chromatography (on Al<sub>2</sub>O<sub>3</sub>) of a fraction of oil of wormwood, b.p. 62-100°, yielded an orange bicyclic oil of wormwood, with 4 double bonds. C<sub>11</sub>H<sub>16</sub>, b.p. 127°, d<sub>4</sub> 0.8826, n<sub>D</sub><sup>20</sup> 1.5864. VIII. The constituents of carotol. 3. A new synthesis of 1,7-dimethyl-4-isopropylphthalimidine. P. Šurma and J. Meláška. Ibid. 96-107.—Carvone subjected to the Reformatskii reaction and followed by hydrolysis yielded carvacrylic acid, m. 112°, which, after heating 6 hrs. with 10% HCOOH, rearranged to carvacrylic acid, m. 89°, which, converted to the acid

chloride and treated with CdMe<sub>2</sub>, followed by treatment with Zn and BrCH<sub>2</sub>COOMe, gave 3-(2-methyl-8-isopropyl)-2-methyl-3-propene-1-carboxylic acid (IV, m. 165-7°). During the above HCOOH treatment 21% I and KIO<sub>4</sub> were also obtained. 3,5-Dimethyl-8-isopropyl-3,4-dihydro-1(2H)-phthalimide (III) was obtained from the ring closure (by AlCl<sub>3</sub>) of the acid chloride of the hydrogenated form of II. III was reduced by LiAlH<sub>4</sub>, to the ale, which was dehydrogenated to 1,7-dimethyl-4-isopropylphthalimidine, m. 89°. The ultraviolet spectra, picrates, and syntheses of this synthetic sample and the one isolated from carotol (cf. C.A. 42, 7293b; 43, 3978b) were identical. IX. The spectra in the visible and ultraviolet regions of 2,6-dimethylazulene (Knesel, Ibid. 201-8.—Absorption spectra of the subject azulenes are compared and their relationship to other methylazulenes is discussed. P. Šurma and O. M. O. Webb. X. On the synthesis of 2,6-dimethylazulene in C<sub>11</sub>. Et 8-methylazulene was cyclized with Na dust in C<sub>11</sub>. to ethyl 3-methylcyclopentan-6-one-1-car-

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**Synthesis of keto dicarboxylic acids - I, Sorm and J.**  
*Dobroge, Gherman, Cernica, Chiriac, Zamfir, 14,*  
*loc. 12, 1949, in English:  $\text{LiAlC}_2(\text{CH}_2)_2\text{OCOCl}$  (10.1 g.)*  
*added to  $\text{CH}_2\text{N}_2$  in ether with cooling and stirring, let*  
*stand 2 hrs., the ether distilled under vacuum, and the*  
*resulting diizo ketone in dioxane added with dil. HCl,*  
*let stand 2 hrs., and fractionated under reduced pressure,*  
*gave 89.2%  $\text{HOOC}(\text{CH}_2)_2\text{COCH}_2\text{CH}_2\text{Cl}$ , b.p. 120°, m.p. 25°*  
*(IV). Similarly,  $\text{LiAlC}_2(\text{CH}_2)_2\text{COCl}$  gave 90%  $\text{HOOC}(\text{CH}_2)_2$   
 $\text{COCH}_2\text{CH}_2\text{Cl}$ , b.p. 120-121°, m.p. 10.5-10°, and  $\text{LiAlC}_2(\text{CH}_2)_2\text{COCl}$   
 $\text{MeCH}_2\text{COCl}$  gave 88%  $\text{HOOC}(\text{CH}_2)_2\text{COCH}_2\text{CH}_2\text{COCl}$ ,  
 $\text{CH}_2\text{Cl}_2/\text{H}_2\text{O}$ , b.p. 131-2°. I and  $\text{CH}_2\text{N}_2$  (10 g.) in  $\text{CH}_2\text{Cl}_2$ ,  
*at room temp. 18 hrs., refluxed 6 hrs., poured into H<sub>2</sub>O,*  
*cooled with  $\text{CaH}_2$ , and the cyst dried over  $\text{Na}_2\text{SO}_4$ , and then*  
*distilled, gave 10%  $\text{HOOC}(\text{CH}_2)_2\text{COCH}_2\text{CH}_2\text{OH}$  (V).*  
*IV, b.p. 118-2°, similarly, II gave 40%,  $\text{HOOC}(\text{CH}_2)_2\text{COCH}_2\text{CH}_2\text{COCl}$ ,  
 $\text{CH}_2\text{Cl}_2/\text{H}_2\text{O}$  (10 g.), (VI), b.p. 101-200°. IV, refluxed 6 hr.*  
*with 10 ml. concd. HCl and 20 ml. glacial AcOH,*  
*evapd. to dryness under a vacuum, ether added, and the*  
*ether-insol. material recrystallized from H<sub>2</sub>O gave 45%  $\text{HOOC}(\text{CH}_2)_2\text{CO}(\text{CH}_2)_2\text{COCl}$ , m.p. 110.5-111°; similarly, V gave*  
*15%,  $\text{HOOC}(\text{CH}_2)_2\text{CO}(\text{CH}_2)_2\text{COCl}$ , m.p. 110.5°. III and*  
*CH<sub>2</sub>Cl<sub>2</sub>/CO<sub>2</sub>/H<sub>2</sub>O in  $\text{CH}_2\text{Cl}_2$  refluxed 1 hr., cooled, poured onto*  
*H<sub>2</sub>O, the top layer acidified, the oil sepr. evapd. with ether,*  
*and the ether evapd. gave  $\text{O}_2\text{CO}(\text{CH}_2)_2\text{CM}_2\text{CH}_2\text{COCl}$ ,*  
*m.p. 133°, subliming at 105-107 mm Hg.**

Herman Skolnik

CA

Terpenes. XI. Infrared investigations of terpenes  
I. J. Pliva and F. Sorm. Collection Czechoslov. Chem.  
Commun. 14, 274-80 (1939) (in English). A comparison  
of the infrared absorption spectra of the hydrogenated  
products obtained from several known types of terpenes  
with the spectra of the original compds. was used in the  
detr. of the C skeleton and constitution of several newly  
isolated sesquiterpenes. Infrared absorption-spectrum  
curves are given for hexahydromyrcene (2,6-dimethylheptane),  
hydrogenated natural farnesene, octahydrofarnesene  
(farnesane), myrcene, natural farnesene, farnesol, deca-  
hydro 8-guaiaulene, tetrahydroguaiene, tetrahydro-4-  
guaiene, guadene,  $\delta$  guaiane, decahydrochamazulene, deca-  
hydrochamazulenogen, chamaulene, chamazulenogen,  
tetrahydro- $\beta$ -caryophyllene, tetrahydrohumulene,  $\beta$  car-  
yophyllene, and humulene. All compds. are carefully  
purified and their  $\text{D}_2^{\text{o}}$ ,  $\text{S}_{\text{D}}^{\text{o}}$  and  $[\alpha]_D^{\text{o}}$  are tabulated.

P. M. Downey

75

**Synthesis of pseudoconhydrine. I. Synthesis of one isomeride of racemic 2-propyl-5-hydroxypiperidine.** I. Senn and J. Sicher. *Collection Czechoslov. Chem. Commun.* 14, 331-341 (1949) (in English). Oxidation of 2-alkyl-hydroxyquinoxalines with fuming HNO<sub>3</sub>, in the presence of Al<sub>2</sub>O<sub>3</sub>, gives 2-alkylquinoxaline acids from which 3-substituted 2-alkylpyridines and piperidines can be prepared. Alkylation of 8-methoxyquinoxaline with PrI gave 2-propyl-8-methoxyquinoxaline (I), b.p. 116-21°/1 mm (acetate, m. 152-3°). Refluxing with 10% HgBr formed 2-propyl-8-hydroxyquinoxaline (II), b.p. 89-91° (acetate, m. 133-0°); benzoate, m. 73-4°. Oxidation gave II', 2-propyl-3,6-pyridinedicarboxylic acid (III), m. 112-3°, and 2-propyl-6-pyridone-5-carboxylic acid, m. 169°. Decarboxylation in boiling BaCO<sub>3</sub>H gave 2-propyl-5-pyridinecarboxylic acid, m. 129-30°, which was converted via the Et ester, b.p. 135-6°, hydrate, m. 90-1°, azide, and urethane, m. 70°, to 2-propyl-5-aminopyridine, b.p. 134-6° (picrate, m. 161°). Diazotization yielded 2-propyl-5-hydroxypyridine, m. 91-2°, which was hydrogenated (Pt catalyst) to *d*-2-propyl-5-hydroxypiperidine, m. 90-1°. Oxidation of 2-propylquinoxaline (IV) with 10% HgBr in H<sub>2</sub>SO<sub>4</sub> and CuSO<sub>4</sub> formed 2,3,4-trimeticarboxylic acid and not the expected III. II can be obtained from IV via the sulfonic acid in poor yields. John Howe Scott

*Terpenes. XVI. Bicyclic sesquiterpene and a new azulene from the oil of *Pogostemon patchouli*.* F. Sorm, L. Dolejš, O. Kressl, and J. Blva. *Collection Czechoslovak Chem. Commun.*, 15, 82-96 (1950) (in English); cf. C.A., 44, 9384a. The constitution of the sesquiterpene hydrocarbons of the oil is investigated. Two fractions chromatographically sept. correspond to tricyclic (I) and bicyclic (II) sesquiterpenes. I, b.p. 112°,  $\delta^{\text{D}}$  = 70.0°, showed one double bond by Pt-H-AcOH. II, b.p. 128°,  $\delta^{\text{D}}$  8.28°, showed 2 double bonds with Pt-H-HOAc. I did not produce an aromatic hydrocarbon by S dehydrogenation, while II yielded S-guaiazulene. II is probably a mixt. of IIa and IIb, as indicated by quant. ozonization products.

MR<sub>D</sub> = 67.25, presumably with shift of an alkyl group  
**XVII. Synthesis of a hydrocarbon of the ionone series**  
 F. Sorm and L. Dolejš. *Ibid.* 96-8. Humulene (I), previously identified as a monoterpenic sesquiterpene, occurs in oil of hops with farnesene and  $\beta$ -carophyllene. The hexahydro deriv. of I has different properties from 3-methyl-1,2,2,6-trimethylcyclohexyl pentane, prep'd. from  $\beta$ -ionone by the Grignard reaction, dehydration, and hydrogenation.

N. T. Furukawa

(IIA)                            (IIB)

The position of the nuclear double bond is uncertain. II is designated 4-guaiazulene. Pt-C dehydrogenation yielded iso-guaiazulene (III). C<sub>14</sub>H<sub>16</sub>, b.p. 118°,  $\delta^{\text{D}}$  1.4918, d<sup>25</sup> 0.8830.

**Condensation of ethyl cyanoacetate with some cyclic anhydrides and imides.** D. Sami, J. G. and P. K. Krušek† (Tech. Univ. Prague) *J. Polym. Sci., Part A: Chem. Commun.*, 15, 391 (1976) (English).  
Methylsuccinimide reacts with the sodium salt of  $\text{NaC}_2\text{H}_3\text{COOC}_2\text{Et}$  to give 60% of the di-ester,  $\text{H}_3\text{C}(\text{CH}_2)_2\text{COOC}_2\text{Et}$ , and 38% of the same product,  $\text{H}_3\text{C}(\text{CH}_2)_2\text{COOC}_2\text{Et}$ , treated with dry HCl gives the di-ester,  $\text{III}$ , and  $\text{I}$  with  $\text{FeCl}_3\text{OEt}_2\text{CH}_2\text{Cl}_2$  to give  $\text{III}$ ,  $\text{III}'$  and  $\text{IV}$ .  
with Adams catalyst in  $\text{CH}_2\text{Cl}_2$  gives the di-ester,  $\text{hydroxyadipate}$ , in 30% yield, and with  $\text{FeCl}_3\text{OEt}_2\text{CH}_2\text{Cl}_2$  gives 41% of the half-ester, in 1.1% yield, and 1.1% pimelic acid, converted by treatment with  $\text{FeCl}_3\text{OEt}_2\text{CH}_2\text{Cl}_2$  into the di-Et ester,  $\text{I}$ ;  $\text{II}$ ,  $\text{III}$  and  $\text{III}'$  condense with Phthaloyl chloride and form  $\text{IV}$ .

II - 4

C.A.

Isolation of hydroxylysine from a gelatin hydrolyzate  
F. Sym and O. Miks. *Collection Czechoslov. Chem. Commun.*, 15, 289-94 (1950) (in English).—Hydroxylysine (I) was obtained from a gelatin hydrolyzate by the application of paper chromatographic and cation exchange procedures, and characterized as the picrate, m. 227-8°. The gelatin hydrolyzate was freed from aromatic amino acids by chromatography on active charcoal and the aliphatic amino acids in the filtrate were absorbed on a column of "Kates Fim Extra." Acidic and neutral amino acids were eluted partially by  $H_2O$  and completely by 2%  $C_6H_5N$ , leaving arginine, I, and lysine (II) which could be eluted with 0.1 N aq.  $NH_4OH$ . Good sepn. of I and II on paper chromatograms could be obtained with  $BuOH-AcOH$  as the mobile phase, with the  $R_f$  values (compared to  $CH_3NHCO_2H$ ) being I, 0.33, and II, 0.21. Bernard Klein

11-4

C.A.

Water-soluble dinitrophenyl derivatives of proteins  
P. Šorga, J. Körbl, and L. Matoulek (Tech. Univ., Prague)  
*Collection Czechoslov. Chem. Commun.*, 15, 283-302 (1950);  
(in English).—H<sub>2</sub>O-sol. dinitrophenyl (DNP) derivs. of  
horse serum albumin, and ovalbumin were prep'd. in a man-  
ner to prevent denaturation, by eliminating the use of  
EtOH and working in NaHCO<sub>3</sub> soln. DNP serum albumin  
was obtained in cryst. form.                   Bernard Klein

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Samm.

*[Handwritten notes]*  
✓ Soviet biochemistry. [P. Šonc, Časopis Lékařů Českých  
90, 757-80 (1981).—A review of the history and recent  
organization of Soviet biochemistry. Anthony Ženíšek]

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SORM, F.

bioactive 1-oxido-alkylidene. III. Synthesis of 1-oxido-alkylidene-  
cyclopropanes and their biological activity. C. R. Acad. Sc. Paris, 267, 1375-1378  
(1968). In this paper, the synthesis of 1-oxido-alkylidene-cyclopropanes is described. These compounds are formed by the reaction of a cyclopropane derivative with a carbonyl compound in the presence of a base. The reaction is carried out in the presence of a base such as sodium methoxide or potassium hydroxide. The resulting 1-oxido-alkylidene-cyclopropanes are then isolated and purified. The biological activity of these compounds is evaluated by testing them against various microorganisms and animal cells. The results show that some of these compounds exhibit significant biological activity, particularly against certain types of bacteria and fungi.

IV. Preparation of 1-oxido-alkylidene-cyclopropanes. C. R. Acad. Sc. Paris, 267, 1379-1382 (1968). In this paper, the synthesis of 1-oxido-alkylidene-cyclopropanes is described. These compounds are formed by the reaction of a cyclopropane derivative with a carbonyl compound in the presence of a base. The reaction is carried out in the presence of a base such as sodium methoxide or potassium hydroxide. The resulting 1-oxido-alkylidene-cyclopropanes are then isolated and purified. The biological activity of these compounds is evaluated by testing them against various microorganisms and animal cells. The results show that some of these compounds exhibit significant biological activity, particularly against certain types of bacteria and fungi.

BA

A3

W-611). The synthesis of (+)-chamomilein D-  
acetophenone, which is a new and of related compounds  
which have been reported to have some  
biological activity on them and on certain  
pathogens. There is no evidence at this time of  
any action of CH<sub>3</sub>OH or O<sub>2</sub> on CH<sub>3</sub> acetophenone.  
CH<sub>3</sub>OH or acetone at CH<sub>3</sub>OH or acetone  
activity. Chamomile acetophenone is a  
CH<sub>3</sub>OH on CH<sub>3</sub> acetophenone, the activity is a slight effect. CH<sub>3</sub>OH  
on CH<sub>3</sub> acetophenone is more effective than the corresponding  
acetophenone alone. The same effect is seen  
when Myrcia baccata alone is used in water  
and the same effect is seen when the same  
is used in CH<sub>3</sub>OH, with the approxi-  
mation of 1000 at the D.P. n=12 K.F. yields the following:  
110, 120, 130, 140, 150, 160,  
170, 180, 190, 200, 210, 220, 230, 240,  
250, 260, 270, 280, 290, 300, 310, 320, 330,  
340, 350, 360, 370, 380, 390, 400, 410, 420,  
430, 440, 450, 460, 470, 480, 490, 500, 510,  
520, 530, 540, 550, 560, 570, 580, 590, 600,  
610, 620, 630, 640, 650, 660, 670, 680, 690,  
700, 710, 720, 730, 740, 750, 760, 770, 780,  
790, 800, 810, 820, 830, 840, 850, 860, 870,  
880, 890, 900, 910, 920, 930, 940, 950, 960,  
970, 980, 990, 1000.

over

X

180'.  $\text{CH}_3\text{N}_c\text{COCH}_2\text{Cl}$  with fused  $\text{NaOAc}-\text{AcOH}$  at the bp.  
(4 hr.) yields  $\text{CH}_3\text{N}_c\text{COCH}_2\text{OAc}$  which yields an amine,  $\text{C}_7\text{H}_{11}\text{N}_c\text{O}_2$ ,  
m.p. 117°, which reduced in  $\text{AsCN}$  ( $16.5^\circ\text{C}$ ) yields 2-acetamido-1-  
methylene-3-phenoxypropane, m.p. 118–119°, which on addition to  
 $\text{H}(\text{NO}_2)-\text{H}_2\text{SO}_4$  (swirling) yields the p-nitro-deriv., m.p. 188–189°,  
from which is obtained by hydrolysis with 5% HCl (water-bath,  
5 hr.) the hydrochloride of 2-amino-3-p-nitrophenoxypropano-1-  
 $\text{N}_c$ ,  $\text{C}_7\text{H}_{11}\text{N}_c\text{O}_2\text{HCl}$ , m.p. 171–172°. [66 Ammonium deriv.  
 $\text{C}_7\text{H}_{11}\text{N}_c\text{O}_2\text{H}_2$ , m.p. 189°, by reaction of  $\text{CHCl}_3-\text{CO}_2\text{Me}$  at 80°  
(3 hr.).] J. G. M. CAMPBELL

CA

Terpenes. XXIII. The composition of oil of carrot (*Ducus carota*). F. Šírov, M. Zaoral, J. Arbat, J. Pliva and V. Herout (Central Chem. Research Inst., Prague). Collection Czech. Chem. Commun. 16, 47-56 (1951); cf. C.A. 45, 4744. Oil of carrot, d<sub>4</sub> 0.893, n<sub>D</sub><sup>20</sup> 1.4653, [α]<sub>D</sub><sup>25</sup> -17.8°, of Dutch origin, was fractionated by means of a 40 theoretical-plate column and the fractions obtained subjected to repeated chromatographic steps. Its qual. compn. as detd. by infrared exams. and the preps. of various derivs. showed that carotol was the principal component of the oil; α- and probably β-phene, dipentene, p-cymene, carvone, geranyl acetate, β-caryophyllene, bergamotene, and bisabolene were other components; a sesquiterpene aldehyde, CuH<sub>10</sub>O, was also isolated. The highest-boiling fractions contained a mixt. of diterpenic hydrocarbons and daucol W. M. Potts

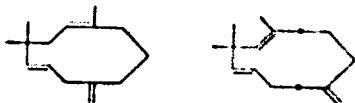
CA  
11A

*Proteins and amino acids. VII. Clupein.* F. Sorm and Z. Šormová (Central Chem. Research Inst., Prague). *Collection Czechoslov. Chem. Commun.*, 16, 207-13 (1951). (in English); cf. C.A. 46, 153c.—Amino acid compns. of the clupein methyl ester hydrochloride fractions prepd. after Felix and Ditt (C.A. 24, 376) were found to vary in amino acid content. Fraction B was found to differ from fractions A and C in that it contained small amts. of aspartic and glutamic acids in addn. to the amino acids common to all fractions. The end groups of clupein were investigated by prep. dinitrophenylclupein (I) as the methyl ester hydrochloride after Sanger (C.A. 46, 5394). The complete hydrolysis of I and analysis of the products on a silica-gel column indicated proline and probably serine as terminal amino acids. Partial hydrolysis of I yielded some neutral dinitrophenyl peptides identified as dinitrophenylprolyl-alanine, dinitrophenylprolylalanylserine, and dinitrophenyl-serylalanylserine. Proline-alanine-serine is indicated as the terminal amino-acid sequence in clupein counter to currently noted formulas for clupein. J. M. Perri

10

CA

The structure of humulene. P. Švec, M. Strebl, J. Pilva, and V. Herout (Central Chem. Inst., Prague, Czech). *Chem. Listy* 45, 309 (1951).—On the basis of hydrogenation, ozonization, mol. refraction (mol. depression -0.6 for D line), and optical inactivity, 2 formulas having 11-membered rings are suggested for humulene:



M. Hudlický

CA

112

Antitubercular activity of 2-amino-4-hydroxy-5-pyrimidinocarboxylic acid, pyrimidine analog of  $\beta$ -amino salicylic acid. P. Sorm, J. Hlavnicka, J. Sieber, and A. Simek (Central Chem. Inst., Prague, Czech.). *Chem. Listy* **45**, 422-5 (1951). - Guanidine carbonate (4 g) in 80

KOH (2.11 g. in 10 ml.) was treated with EtOCH<sub>2</sub>CO<sub>2</sub>H. Crystals of Et-2-amino-4-hydroxy-5-pyrimidinocarboxylate were sepd. and recrystd. from water with a small amt. of EtOH or from AcOH, yield 1.3 g. (62%), m. 230° decomp.<sup>1</sup> The free acid was obtained from its ester by sapon. with ethanolic KOH and acidification with AcOH in a 76% yield, m. 230° (decomp.). Its Na salt crystallizes with 2 H<sub>2</sub>O. Tuberculostatic activity of this acid is half that of  $\beta$ -aminosalicylic acid. M. Hudlický

SOMI F.

O sovotske biochemii. Biochemistry in the Soviet Union  
Cas. lek. cesk. 90:25 22 June 51 p. 757-60.

1. Names of institutes and directors given, also their respective fields of research.

CLML 20, 10, Oct. 51

"APPROVED FOR RELEASE: 08/25/2000

CIA-RDP86-00513R001652420013-7

SCRN, Frantisek

Chemical Abst.  
Vol. 48  
Apr. 10, 1954  
General and Physical Chemistry

3  
Laboratorial chemické předpisy. Edited by Frantisek  
Frum, Prague, 1952. 88 pp. 28 Kčs. Reviewed in  
Chem. Listy 47, 630 (1953).

APPROVED FOR RELEASE: 08/25/2000

CIA-RDP86-00513R001652420013-7"

*organic chemistry - 10*

Separation of dinitrophenylhydrazones by chromatography on activated calcium sulfate. F. Šurán, M. Suchý, and V. Hlavout (Central Chem. Inst., Prague, Czech.) *Chem. Listy* 60, 55-6 (1966).—A mix. of 2,4-dinitrophenylhydrazones was successfully sepd. by chromatography of a  $\text{CaSO}_4$  soln. on  $\text{CaSO}_4$ , freshly ppzd. from  $\text{CaCl}_2$  and  $\text{H}_2\text{SO}_4$  and dried 7 hrs. at 100-110°. Elution was carried out with petr. ether under a pressure of 200 mm. M. Hudlický

General & Physical  
Chemistry L 2

The formal titration of thiazolidine-4-carboxylic acid.  
L. Matoušek and F. Šurm (Central Chem. Inst., Prague,  
Czech.), *Chem. Listy* 46, 111-12 (1952).—Math. treatment  
of equil. of  $\text{CH}_3\text{O}$  and hydroxylamino acids and mercapto-  
amino acids led to a value of 5.03 for the dissoci. const. of  
methylthiazolidine-4-carboxylic acid. M. Hudlický

SORM, František  
František Sorm

Proteins and amino acids. XIII. Use of azobisisocyanate-sulfonyl chloride in the determination of end amino acids of peptide chains. Bohumil Kell, Věra Kneaslová, and František Sorm. Chem. Listy 46, 167-70(1952).—The journal reference in U.S.A. 48, 3904c should have been Chem. Listy 46, 167-70(1952) instead of Ibid. 167-70. B. J. C.

Handwritten notes - +

Isolation of a spasmolytic substance from Matricaria chamomilla. F. Šejn, Z. Čekan, V. Herout, and H. Raková (Central Chem. Inst., Prague, Czech). *Chem. Listy* **66**, 308 (1952). —The spasmolytic principle of chamomile, *apigenin* (I) (0.45 g.), was isolated by digestion of chamomile biomass (2.844 g.) followed by ether extn. The triacetyl deriv., m. 178-81° (from EtOH), prep'd. from 120 mg. I and 7.5 ml. Ac<sub>2</sub>O was hydrolyzed with 10% HCl to give a yellow powder, m. 232-4°. M. Hudlický

Biological Chemistry  
II

Properties of transamidase. František Šurina, Karel Šebesta, and Tomáš Turský (Central Chem. Inst., Prague, Czechoslovakia). *Chem. Listy* 46, 373-4 (1952).—Kidney transamidase was found to be a lysozyme which does not require the presence of a specific coenzyme. The enzyme is inhibited by heavy metals and by ornithine, but not inhibited by CN<sup>-</sup> and Complexone. The enzyme is stable at pH 5.0 and is inactivated by heating 5 min. at 30°. Activity of the transamidase was followed by paper chromatography, which separates arginine from glycocyanine. Ornithine was found among the products of the enzymic reaction of arginine and glycine.  
M. Hudlický

*Biochemical Chemistry. A  
General II*

**Proteins and amino acids. XIII. Activation of chymotrypsinogen to chymotrypsin.** Frantisek Sorm, Bohuslav Karel, and Ivan Rychlik. (Central Chem. Inst., Prague, Czech.). *Chem. Listy* **46**, 401-4 (1952); cf. *C.A.* **46**, 11294Y. - On the basis of quant. paper chromatography of dinitrophenyl derivatives of amino acids, chymotrypsinogen (I) was found to contain no basic end group. Activation is followed by the formation of 2 amino groups (based on the mol. wt. 22500). In addition, a mixt. of tri- to octapeptides is formed as a result of hydrolytic processes. Cryst.  $\alpha$ -chymotrypsin consists of 2 or 3 proteins having the same proteolytic activity. During the crystn., the content of a form contg. 1 mole of alanine and 1 mole of phenylalanine as end amino acids increases. Proteins with aspartic acid, serine, and threonine as end amino acids accumulate in the mother liquors. Activation of I is based probably on the cleavage of cyclic peptide chain which reveals the center of activity. XIV. Enzymatic activity of dinitro derivatives of  $\alpha$ -chymotrypsin. Frantisek Sorm and Ivan Rychlik. *Ibid.* **46**, 8. - By the action of 1,2,4-C<sub>6</sub>H<sub>3</sub>(NO<sub>2</sub>)<sub>3</sub> 150 mg. on lyophilized chymotrypsin (I) (300 mg. in a soln. contg. 300 mg. NaHCO<sub>3</sub> in 15 ml. H<sub>2</sub>O), dinitrophenyl derivs. contg. 2, 4, and 7 dinitrophenyl

groups per mol. of I, resp., were prep'd., purified by dialysis and analyzed by spectrophotometry. Protease and chymotrypsin activities drop with increasing rate of substitution; esterase activity of I contg. 2 dinitrophenyl group is higher than contg. 4 groups about the same, and that contg. 7 dinitrophenyl groups per mol. of I lower than that of I. Michaelis const. and max. reaction rate of I pure and I contg. 2, 4, and 7 dinitrophenyl groups, resp., are: 0.013, 0.0012, 0.0023, and 0.0018; 0.39, 0.40, 0.39, and 0.12. Tendency of synthesizing polypeptides of methionine from its Pr ester increases with increasing amt. of dinitrophenyl groups. XVI. Interaction of proteins with electrolytes. Vladimir Kacera and Lubek Matousek. *Ibid.* **46**, 323-8; cf. *C.A.* **46**, 1131A. - A dynamic equil. exists in a system protein-metal ion in which protein (serum albumin) represents a complexing agent of low diffusion const. This accounts for the fact that a wave showing the reduction of ions forming a complex with serum albumin is, at a certain pH, higher than that corresponding to the flow of ions in the complex. M. Hindlekt

Histochemistry - 11c  
v6

Proteins and amino acids. XV. Isolation and properties  
of protein toxins of *Shigella dysenteriae*. František Švarc,  
Zora Švarcová, Pavla Šebestová, and Věra Matějková  
(Central Chem. Inst., Prague, Czech). *Chem. Listy* 46,  
468-70 (1952); cf. *C.A.* 46, 11204d. Ext of the R strain of  
*Shigella dysenteriae* (Shiga-Krusae) was fractionally precipitated  
with  $(\text{NH}_4)_2\text{SO}_4$ , and the concentrate subjected to electrophoresis  
in an agar gel. Out of 3 protein components, the  
two prevailing were found extremely toxic, having  $\text{LD}_{50}$  0.3  
and 0.075 γ for 18 g. mice, resp. The more toxic com-  
ponent contains β-alanine. Both toxins seem to be pure  
proteins, contg. no nucleic acids. M. Hudlický

Biological Chemistry  
General - II d

CA

Identity of the green liver protein with catalase. E.  
Sorm and K. Šebesta (Central Chem. Inst., Prague, Czech.).  
*Chem. Listy* 46, 309 (1952).—On the basis of infrared spec-  
tra, the green protein isolated from horse liver by Lauf-  
berger (*Anal. Listy* 22, 200 (1937)) was found to be identical  
with catalase from horse liver. M. Hudlický

NEJEDLY, Zdenek; SORM, F.

Telegram of the Czechoslovak Academy of Sciences expressing  
condolences on the death of Generalissimo Iosif Vissarionovich  
Stalin, President of the Council of Ministers. Chekh.biol. 2  
no.1:3 Ap '53.

(MLRA 7:2)

1. Prezident Chekhslovatskoy Akademii nauk (for Nejedly).
2. Glavnyy sekretar' Chekhslovatskoy Akademii nauk (for Sorm);  
(Stalin, Iosif, 1879-1953)

SORM, F.

NEJEDLY, Z.; SORM, F.

Telegram of condolence sent by the Czechoslovak Academy of Sciences on the occasion of the death of Generalissimo I.V. Stalin, Chairman of the Council of Ministers [in Russian and English]. Chekh.fiz.shur. 3 no.1; 1,7 Mr '53.  
(MLRA 7:6)

1. President of the Czechoslovak Academy of Sciences (for Nejedly).
2. General Secretary of the Czechoslovak Academy of Sciences (for Sorm).  
(Stalin, Iosif, 1879-1953)

*✓ Advancements in the chemistry of sesquiterpenes.*  
Ch František Sorm *XIIth Intern. Congr. Pure Appl. Chem., Szczecin 1951; Collection Czechoslov. Chem. Commun., Suppl. 2, 19, 68-80 (in German); Magyar Tudományos Akad. Kemiai Tudományos Osztályának Kiadványai 3, 351-60 (1953); cf. C.A. 47, 9044c.*—Sesquiterpene hydrocarbons were isolated and identified by distn. combined with chromatographic adsorption on alk.  $\text{Al}_2\text{O}_3$  and infrared spectrophotometry. It is possible to sep. sesquiterpenes differing in no. of double bonds or in C skeletons. The infrared spectra of caryophyllene from clove oil, oil of wormwood, and oil of bergamot were compared with the spectra of  $\gamma$ -cadinene and  $\delta$ -cadinene from oil of citronella,  $\epsilon$ -cadinene from ylang-ylang oil, cadinene regenerated from  $\epsilon$ -cadinene-HCl, tetrahydro- $\gamma$ -cadinene, tetrahydro- $\delta$ -cadinene, and cadinane from regenerated cadinene. The explanation of structures of unknown compds. was aided by hydrogenation and elimination of functional groups of the sesquiterpenes in order to compare the skeleton with known std. hydrocarbons. By this method the structures of 2 diterpene hydrocarbons,  $C_{16}\text{H}_{26}$  and  $C_{16}\text{H}_{24}$ , isolated from oil of wormwood were clarified. The infrared spectra of the partially hydrogenated aromatic diterpene,  $C_{16}\text{H}_{24}$ , were shown to be similar to that of 2,6-dimethyl-10-( $\alpha$ -tolyl)-undecane. The structures of monocyclic sesquiterpenes of the bisabolane, humulene, and elemol (I) type were discussed. The skeleton of I appears to be 1-methyl-1-ethyl-2,4-diisopropylcyclohexane. Dicyclic sesquiterpenes such as cadinene, calacorene, caryophyllene, caratol, chamazulene, and the isomeric acorone and isoacorone (from calamus oil) were discussed and structures suggested for them.

M. M. Bender

MATOUSEK, L.; SORM, F.

On proteins and aminoacids. Part 11. On clupein. Part 2. [in German  
with summary in Russian]. Sbor.Chekh.khim.rab. 18 no.1:1-6 1953.  
(MIRA 7:6)

1. Tsentral'nyy khimicheskiy institut, Praga. (Clupein)

*20K-1-1*  
SICHER, J.; PARKAS, J.; SOEM, F.

Studies in the chloramphenicol series. Part 4. Synthesis of 1-(p-nitro-phenyl)-2-hydroxymethyl-2-dichloro-acetamido-1,3-propandiol and a correction [in English with summary in Russian]. Sbor.Chekh.khim.rab. 18 no.1:102-105 F '53. (MIR 7:6)

1. Chentral Chemical Research Institute, Prague.  
(Chloramphenicol)

*SOK/M/*

SORM, F.; VERES, K.; HEROUT, V.

On terpenes. Part 36. The constitution of calamenene [in English with summary in Russian]. Sbor.Chekh.khim.rab. 18 no.1:106-115 P '53.  
(MLRA 7:6)

1. Central Chemical Research Institute, Prague.  
(Sesquiterpenes)

*10/17/74*  
SORM, F.; ZAORAL, M.; HROUT, V.

On terpenes. Part 38. On the constitution of natural bisabolol and  
bisabolol monoxide from matricaria oil [with summary in English].  
Sbor.Chekh.khim.rab. 18 no.1:116-121 P '53. (MLRA 7:6)

1. Central Chemical Research Institute, Prague.  
(Bisabolol) (Matricaria oil)

*SORM, F.*  
HEROUT, V.; ZAORAL, M.; SORM, F.

On terpenes. Part 39. Synthesis of two tetrahydrobisabolols [with summary in English]. Sbor.Chekh.khim.rab. 18 no.1:122-126 P '53. (MLRA 7:6)

1. Central Chemical Research Institute, Prague.  
(Bisabolol) (Matricaria oil)

*Dvihy, F.*  
SOHM, F.; CEKAN, Z.; HEROUT, V.; RASKOVA, H.

Isolation spasmolytically active substance from Matricaria chamomilla L. [with summary in English]. Sbor.Chekh.khim.rab. 18 no.1:127-130 P '53.  
(MLRA 7:6)

1. Central Chemical Research Institute, Prague. 2. Institute of Pharmacology of the Medical Faculty, Charles University, Prague.  
(Matricaria oil) (Flavones)

*DOX-1*  
SMRT, J.; SORM, F.

Proteins and amino-acids. Part 12. Synthesis of  $\alpha$ -methylglutamic acids  
[with summary in English]. Sbor.Chekh.khim.rab. 18 no.1:131-139 F '53.  
(MLRA 7:6)

1. Central Chemical Research Institute, Prague.  
(Methylglutamic acids)

SORM, F.; SEBESTA, K.; TURSKY, T.

Some properties of transamidinase [with summary in English]. Sbor.  
Chekh.khim.rab. 18 no.1:140-150 F '53. (MIRA 7:6)

1. Central Chemical Research Institute, Prague.  
(Transamidinase)

*SORM, F., KEIL, B., RYCHLIK, I.*

On albumins and aminoacids. Part 15. Activation of chymotrypsinogen to chymotrypsin [with summary in German]. Sbor.Chekh.khim.rab. 18 no.2: 285-293 Ap '53. (MLRA 7:6)

1. Institut organicheskoy khimii Chechhoslovatskoy Akademii nauk, otdeleniye organicheskoy biokhimii, Praga. (Chymotrypsin)

SORM, F.; CERNY, V.

"Steroids. IV. New Method for Preparation of 3-Hydroxyetienic-(5) acid from  
3-acetoxy-17-ketoandrostan-(5)." P. 407  
(COLLECTION OF CZECHOSLOVAK CHEMICAL COMMUNICATIONS. SBORNÍK ČESkoslovenských  
KHEMICKÝCH RABOT. Vol. 18, No. 3, June 1953-Praha, Czech. )

So: Monthly List of East European Accessions, (EEAL), LC, Vol 4, No. 4,  
April 1955, Uncl.

"APPROVED FOR RELEASE: 08/25/2000

CIA-RDP86-00513R001652420013-7

✓ Proteins and amino acids. XIV. Enzymic activity of  
dinitro derivatives of  $\alpha$ -chymotrypsin. F. Šorm and I.  
Rychlík. Collection Czech. Chem. Commun. 48, 413-21  
(1983).—See C.A. 46, 11264f.

H. L. H. /

APPROVED FOR RELEASE: 08/25/2000

CIA-RDP86-00513R001652420013-7"

Proteins and amino acids. XV. Isolation and properties  
of protein toxins of *Shigella dysenteriae*. F. Šorm, Z.  
Šonarová, P. Šebestová, and V. Matějovský. *Czechoslovak  
Czech. Chem. Commun.* 18, 423-7 (1953). See C.A. 46,  
113164. H.L.H.

SEARCHED INDEXED  
SERIALIZED FILED

✓Chloramphenicol series. V. Analogs containing chlorine in the side chain and oxazolines. Jiri Pařík and Jiří Sicher. (Czech. Akad. věd, Prague). Collection Czechoslov. Chem. Commun. 18, 560-564 (1963) (in English).—See C.A. 49, 2182. VI. Stereo course of the reduction of deshydrochloramphenicol and related compounds. Jiří Sicher, Miroslav Svoboda, Magdalena Hrdlù, Josef Rüdinger, and František Šurán (Czech. akad. věd., Prague). Ibid. 487-490. See C.A. 49, 2186. H. L. H.

Sorm, F.

SICHER, J.; SVOBODA, M.; HRDA, M.; RUDINGER, J.; SORM, F.

Studies in the chloramphenicol series. Part 6. Steric course of the reduction of dehydrochloramphenicol and related compounds [in English with summary in Russian]. Sbor.Chekh.khim.rab.18 no.4:487-499 Ag '53.  
(MLRA 7:6)

1. Department of Organic Synthesis, Institute of Organic Chemistry,  
Czechoslovak Academy of Science, Prague. (Chloromycetin)

SORM, F.

-1205\* On Terpenes. XLIII. Infrared Investigations of  
Terpenes. IV. (English.) L. Plival' V. Herout, B. Schneider  
and F. Sorm /Collection of Czechoslovak Chemical Communi-  
cations, V. 10, no. 4, Aug. 1959, p. 800-811.

Infra-red spectra of cadinanes, tetrahydroszingiberene, and  
calacorane show that these hydrocarbons differ from each other  
merely in their spatial configuration. Tables, graphs. 22 ref.

Inst.-Org. Chem., Czechoslovak Acad. Sci., Prague.

*D.R.A. 5.*  
SORM, F.; HOLUB, M.; SYKORA, V.; MLEZIVA, J.; STREIBL, M.; PLIVA, J.;  
SCHNEIDER, B.; HEROUT, V.

On terpenes. Part 46. Sesquiterpenic hydrocarbons from oil of sweet  
flag [in English with summary in Russian]. Sbor.Chekh.khim.rab. 18  
no.4:512-526 Ag '53. (MLRA 7:6)

1. Department of Natural Products, Institute of Organic Chemistry,  
Czechoslovak Academy of Science, Prague. (Sesquiterpenes)  
(Calamene)

*SORM, F.*

SORM, F.; NOVAK, J.; HEROUT, V.

On terpenes. Part 51. The composition of chamaulene; preliminary communication [in English with summary in Russian]. Sbor.Chekh.khim. rab. 18 no.4:522-529 Ag '53. (MLRA 7:6)

1. Department of Natural Products, Institute of Organic Chemistry, Czechoslovak Academy of Science, Prague.  
(Chamaulene)

SEARCHED, INDEXED.

Amino acids and peptides. VIII. Peptides of 2,4-di-  
aminobutyric acid. Milan Zentral, Josef Radliger, and  
Frantisek Sorm (Czech. Acad. Sci., Prague). Collection  
of Czechoslovak Chemists 19, 630-45 (in Russian). Eng.  
summary, 649-9 (1953).—See C.A. 49, 170a, 13.  
Constitution of phallolidine. 2. Bedrich Meloun, Bohuslav  
Keil, and Frantisek Sorm. *Ibid.* 19, 153-60 (1954) (in  
German).—See G.A. 49, 180d. H. L. H.

SOKR, F.

KBIL, B.; SOKR, F.

On proteins. Part 19. Methylation of chymotrypsogen and chymotrypsin  
[with summary in English]. Stor.Chekh.khim.rab. 18 no.4:550-559 Ag '53.  
(MLRA 7:6)

1. Department of Organic Biochemistry, Central Chemical Research Institute,  
Prague. (Chymotrypsin) (Methylation)